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ERDC/CERL TR-08-5, Vol. 1

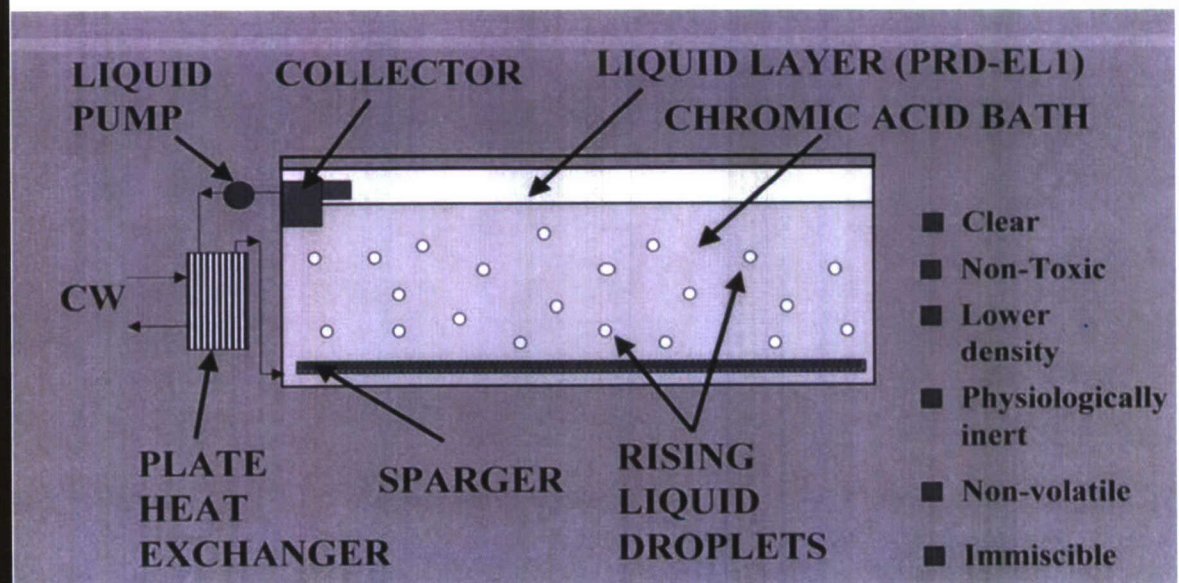
Construction Engineering
Research Laboratory

Technology Demonstration of the Zero Emissions Chromium Electroplating System

K. James Hay, Stephen W. Maloney,
John J. Cannon, Max R. Phelps, and
Jason Modrell

February 2008

20080506216



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K. James Hay and Stephen W. Maloney

*U.S. Army Engineer Research and Development Center
Construction Engineering Research Laboratory
PO Box 9005
Champaign, IL 61826-9005*

John J. Cannon

*Benét Laboratories
Watervliet Arsenal
Watervliet, NY 12189-4050*

Max R. Phelps and Jason Modrell

*Pacific Northwest National Laboratory
P.O. Box 999
Richland, WA 99352-0999*

Final Report

Approved for public release; distribution is unlimited.

Prepared for U.S. Environmental Protection Agency
 26 West Martin Luther King Drive
 Cincinnati, OH 45268-0001

Under Work Unit #CNE-B091

ABSTRACT: This report documents the demonstration of a technology developed by PRD, Inc, for control of chromium emissions during hard chromium electroplating, the Zero Emissions System. The technology involves placing a blanket of a proprietary fluid, called PRD-EL1, on top of the plating bath. This fluid blanket prevents the formation of aerosols, which is the mechanism by which chromium is emitted from the plating bath to the air. The majority of the testing was directed at demonstration of the effectiveness of chromium plating in the presence of the immiscible blanket. Testing was conducted at Benét Laboratories on coupons and actual parts from Army vehicles. The results indicate that PRD-EL1 may cause deleterious effects on the plating process, as some of the parts failed qualitative tests performed at Benét. However, some parts, which were plated without the fluid blanket present as a baseline control, also failed the tests. Air sampling results indicate that the presence of the PRD-EL1 fluid reduced the chromium emissions to below the standard and the indoor air concentration below the previously established exposure limit but near the new exposure limit. Overall, the results indicate that the use of the PRD process would require additional testing before it could be accepted for use in Army production and maintenance operations.

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Conversion Factors

Non-SI* units of measurement used in this report can be converted to SI units as follows:

Multiply	By	To Obtain
degrees Fahrenheit	$(5/9) \times (^\circ\text{F} - 32)$	degrees Celsius
inches	0.0254	meters

* *Système International d'Unités* ("International System of Measurement"), commonly known as the "metric system."

Preface

This study was conducted for Headquarters, Department of the Army, under Program Element 063728A, “Environmental Technology Demonstration”; Project 002, “Environmental Compliance Technology”; Work Unit CNE-B091, “Hazardous Air Pollutants Technology Demonstrations.” This project is part of the Army Environmental Quality Technology (EQT) Program. The ERDC technical reviewer was Hany Zaghloul, Program Manager.

The work was performed by the Environmental Processes (CN-E) Branch of the Installations Division (CN), Construction Engineering Research Laboratory (CERL). The CERL Principal Investigator was Dr. K. James Hay. Part of this work was done by Anniston Army Depot (POC: Tony Pollard), Benét Laboratories (POC: John Cannon), the Center for Health Promotion and Preventive Medicine (POC: Tim Hilyard), and PRD, Inc. (POC: Dr. Ramesh Melarkode). Deborah Curtin is Chief, CN-E, and Dr. John T. Bandy is Chief, CN. Dr. Kirankumar V. Topudurti is Deputy Director of CERL and the Director of CERL is Dr. Ilker R. Adiguzel.

CERL is an element of the U.S. Army Engineer Research and Development Center (ERDC), U.S. Army Corps of Engineers. The Commander and Executive Director of ERDC is COL Richard B. Jenkins. The Director of ERDC is Dr. James R. Houston.

1 Introduction

Background

Chromium plating falls into two categories based on the thickness of the chromium deposit. Decorative or “bright chromium” plating has a plating thickness of $0.80\text{ }\mu\text{m}$ or less and provides a bright, reflective, corrosion-resistant coating for metals and plastics, while functional or “hard chromium” deposits are usually greater than $0.80\text{ }\mu\text{m}$ and are used for industrial purposes such as wear resistance.

Hard chromium plating is used extensively throughout the Department of Defense (DoD) to protect mission-critical military components from wear and corrosion. Although several competing technologies have been considered, the DoD has not found a suitable alternative to chromium plating. This has made hard chromium the standard plating process for protecting these mission-critical components in U.S. Army land- and air-based weapons platforms.

Unlike other plating processes, chromium cannot be directly deposited on the target substrate from an aqueous bath containing the metal ions. The chromium plating process must use at least one acid radical to catalyze the cathodic deposition of the chromium onto the piece being plated, and the chromium is usually supplied through a complex chemical reaction from sources containing either the hexavalent Cr^{+6} or Cr^{+3} ion.

Anodes for the process can be made from a variety of substrates depending on the application, but the preferred anode is made of a lead alloy. Other metals are typically used for more specialized applications. Watervliet Arsenal (WVA) uses a lead/copper alloy, while Anniston Army Depot (ANAD) uses iron anodes.

ANAD, Corpus Christi Army Depot (CCAD), and WVA use the more traditional process whereby chromic acid (CrO_3) is mixed with sulfuric acid (H_2SO_4) and water (H_2O) to provide the plating solution. The electrochemical process reduces Cr^{+6} to elemental chromium for deposition onto the part being plated and, in the process, produces an excess of hydrogen. These hydrogen bubbles form at the cathode and rise through the plating bath to break at the surface of the solution. When the bubbles break, a fine mist of hexavalent chromium is dispersed into the air above the plating bath.

These emissions pose a serious health hazard and are highly regulated. Chromium has long been known to be extremely carcinogenic and long-term use can lead to other medical problems such as perforated nasal passages and persistent skin rashes.

As such a number of different technologies have been developed to either capture the emissions or modify the bubble-bursting action at the surface to prevent chromium emissions (Hay 1996; Hay et al. 1999). The most commonly used method for hard chromium plating operations is to vent the emissions into ductwork and then filter the air through a woven mesh screen (demister) to remove the mist from the air stream and thus collect the chromium.

These demisters work by rapidly changing the direction of airflow through the mesh screen, which causes the mist to disengage from the exhausted air. While in operation, these demisters tend to become clogged with the components of the mist they are removing from the air stream and from the water used to supply the demisters. Demisters and their operations are also heavily regulated and require a minimum airflow through the mesh to operate correctly. As such, any excessive pressure drop across the demister results in a need to either clean or replace the demister. In either case, the operation results in downtime for the plating line and can result in heavy regulatory fines if an excessive concentration of chromium (VI) is released to the atmosphere.

ANAD currently uses demisters in an air ventilation system that collects the emissions from just a few inches above the plating solution surface. They also use polypropylene balls to provide a bursting surface for the bubbles such that any entrained chromium will collect on the ball and return to the bath, which allows for the free escape of hydrogen. While these methods reduce potential chromium emissions by 98 percent or greater, the desire to move to a more efficient, less costly technology is still strong.

A great deal of care must be used in choosing alternative technologies because the hexavalent chromium plating process is sensitive to minor changes in either temperature or current densities. The alternative must be compatible with both the bath and the process. For example, the cathodic current efficiency is inversely proportional to the temperature of the bath, but is directly proportional to the current density. On the plus side, the process is not adversely affected by minor bath contaminations. Emissions-reducing foams or blankets are the more promising alternative technologies; however, care must be given to address the issue of temperature control of the plating bath and to avoid entrapment of the excess hydrogen being produced by the plating process.

The Zero Emission System (PRD Tech, Inc., Florence, KY) uses a proprietary fluid to form an emissions blanket in the form of an immiscible liquid layer that covers the chromic acid bath to eliminate or substantially reduce chromium (VI) aerosol emissions from the tank during plating. Results have been promising on smaller scale demonstrations for reducing emissions resulting from hard chromium plating. The U.S. Army Engineer Research and Development Center/Construction Engineering Research Laboratory (ERDC/CERL) began exploring this technology as an option for Army plating operations in 2000 as part of the ERDC/CERL Hazardous Air Pollutants (HAP) Project.

Objective

The objective of the ERDC/CERL HAP Project is to develop and demonstrate cost-effective technologies to help the Army meet current and future demands of HAP regulations. The primary objective of this study was to determine if the Zero Emission System eliminates chromium emissions from a production-scale chromium plating operation, while not adversely affecting the quality of the plating process. Specifically, the goals were to meet military specifications for plating quality (AMS-QQ-C-320), the standard of 0.015 mg/dscm (milligrams per dry standard cubic meter) for stack emissions set by the Chromium Electroplating National Emission Standard for Hazardous Air Pollutants (NESHAP; Federal Register (FR) 60:4948), and the previous Occupational Safety and Health Administration (OSHA) permissible exposure limit (PEL) for hexavalent chromium [Cr(VI)] of 0.052 mg/m³ of air as an 8-hour time-weighted average (U.S. Code of Federal Regulations [CFR], Title 29, Section 1910). Since the completion of this study, OSHA finalized a new PEL for Cr(VI) of 0.005 mg/m³ of air as an 8-hour time-weighted average (29 CFR 1910, 1915, et al.)

Approach

Data were gathered related to the full-scale implementation of the Zero Emission process, showing that parts plated with this process in place meet military technical specifications for the chromium electroplating finish (AMS-QQ-C-320). Demonstration testing was completed on an active production-scale plating line (Building 114 at the ANAD in Anniston, AL). Selected military parts were plated during two testing phases. The parts were then tested to determine the quality of plating with re-

spect to AMS-QQ-C-320. Air sampling was conducted during the second testing phase to determine if air quality goals could be met.

Mode of Technology Transfer

This technology was installed and tested on a full-scale electroplating tank at ANAD. The technology has since been removed. This report documenting the demonstration testing of the Zero Emission System will be made accessible through the World Wide Web (WWW) at URL: <http://www.cecer.army.mil>.

The HAP project is part of the Army Environmental Quality Technology (EQT) Program. This technology was tested through the ERDC/CERL HAP Project, which uses 6.3 advanced development and field-testing funds. As part of the EQT process, technology transfer plans are being developed by the U.S. Army Environmental Center (AEC) for qualified HAP project technologies.

2 Technology Description

The Zero Emission System developed by PRD Tech, Inc. uses a proprietary immiscible liquid (PRD-EL1) to cover the chromium bath during plating. PRD-EL1 is circulated from the top of the tank down through the liquid sparger assembly. The liquid rises through the chromic acid bath back to the surface, which provides the necessary mixing in the tank to ensure good heat transfer during plating. The layer resting on the top of the bath captures the bubbles produced during plating, preventing them from breaking at the liquid-air interface. By preventing the breakage of bubbles at the liquid-air interface, the process prevents the formation of hazardous chromium aerosols that have traditionally been dealt with through expensive scrubber technology. Figure 1 shows the process flow. Gases entrained in the PRD-EL1 liquid (hydrogen and oxygen) can be separated in a PRD-EL1 liquid storage tank or allowed to pass through the liquid into the existing ventilation system.

The cost of the Zero Emissions System depends on the amount of design, retrofitting, and PRD-EL1 fluid necessary. Chapter 3 lists the cost of the system used in this demonstration.

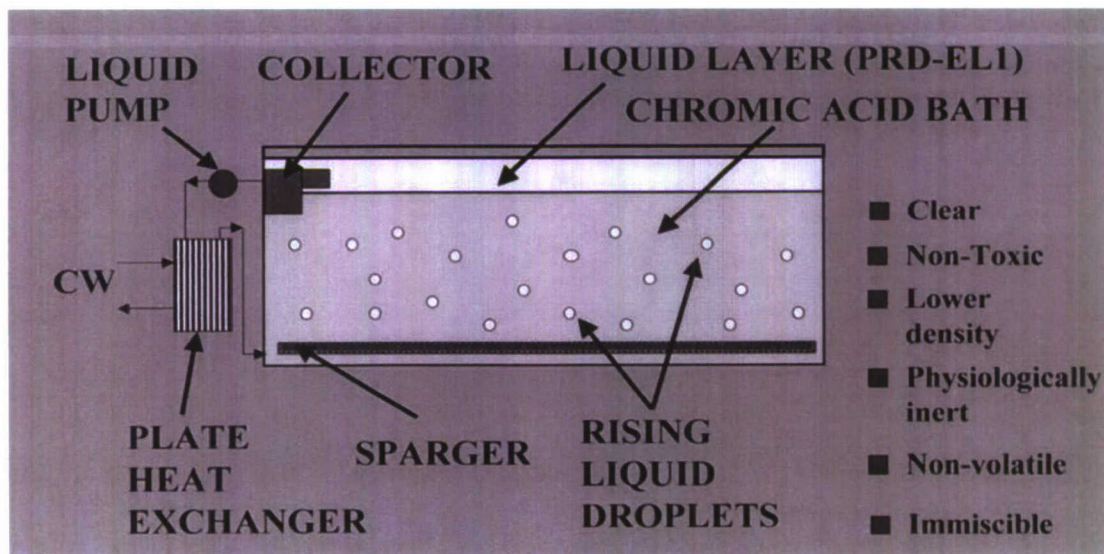


Figure 1. Zero emissions process flow.

3 Site/Facility Description

Site/Facility History

Anniston Army Depot is an active facility occupying 15,200 acres in Calhoun County, AL, about 8 miles west of the city of Anniston. ANAD consists of an ammunition storage area, an industrial area, and an administrative and housing facility. Since 1948 the depot has repaired and modified combat vehicles and artillery equipment.

ANAD is the only Army depot capable of performing maintenance on both heavy and light-tracked combat vehicles and their components. It is the designated Center of Technical Excellence for the M1 Abrams Tank and is the designated candidate depot for the repair of the M60, AVLB, M728, and M88 combat vehicles. ANAD has assumed responsibility for towed and self-propelled artillery as well as the M113 Family of Vehicles. Chromium plating operations at the installation are used to refurbish and manufacture critical components of these combat vehicles.

Under partnership agreements, a wide range of vehicle conversions and upgrades are currently underway. Depot personnel also perform maintenance on individual and crew-served weapons, as well as land combat missiles and small arms. Additionally, the maintenance and storage of conventional ammunition and missiles, as well as the storage of seven percent of the Nation's chemical munitions stockpile (until the stockpile is demilitarized), are significant parts of the depot's overall missions and capabilities.

Site/Facility Characteristics

The site of the Zero Emissions System demonstrations was Building 114, Chromium Plating Line 2, tanks 12-a and 12-b. Building 114 houses the depot's metal finish operations. The building houses two chromium electroplating lines, Lines 1 and 2. Each line has four plating tanks and its own connected ventilation and air pollution control systems. The facility location allows access to the chromic acid bath and drain connections to the building's chromic acid waste lines, electrical power, and sufficient floor space for equipment installation.

Demonstration Setup

The Zero Emissions System was installed on tank 12-b (Figure 2) of Chromium Plating Line 2, Building 114. The process was also installed in two of the remaining three tanks in Line 2 and was operational in tank 12-a during Phase II. However, no test parts were plated in tank 12-a. A sacrificial part was plated for additional current load for the air emissions study that coincided with Phase II. During this demonstration, the system did not require a reservoir tank for the PRD-EL1 liquid; however, space was maintained in case it was needed. Additionally, a liquid sparger was installed in the tank to sparge PRD-EL1 liquid throughout the plating bath. A small pump and other minor plumbing were required to achieve this operation. Table 1 describes the placement of the pumps in the plating tanks.

Implementation Cost

The cost to retrofit one tank (12-b) at ANAD with the Zero Emissions System was \$32,540. This cost included the design, fabrication, installation, and PRD-EL1 to a 4-in. depth. Two additional tanks on Line 2 (12-a and 12-c) were retrofitted with the Zero Emissions System for \$58,850 during the second phase of the demonstration. Tank 12-d was not used as an electroplating tank during this demonstration. Therefore, the total cost to implement this system at ANAD on a plating line of three tanks was \$90,390.

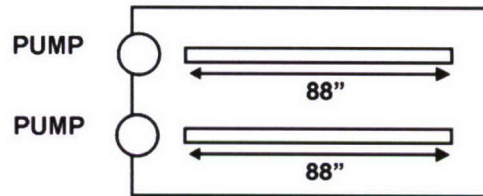
Other implementation issues can affect the total cost of operation for this system. Such issues (e.g., process waste, reliability, ease of use, procurement, and maintenance) were considered during the implementation and testing of the Zero Emission System at ANAD.



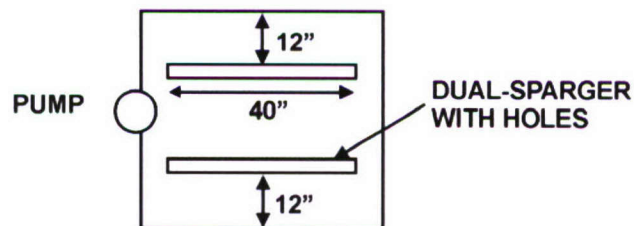
Figure 2. Tank 12-b with the PRD Zero Emissions System installed.

Table 1. Pump placement in tanks.**Tank 12-a****2200 Gallon Tank (96 x 46 x 117 in. deep) with Maximum 12-in. Freeboard**

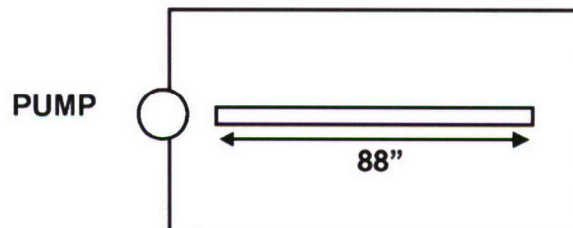
- Two BX3000-18 with Titanium-tip shaft, 90AFR1 adjustable flow restrictor and two PMCXF20 (20-in. long) Permacore Super Magnum Reusable Filters
- Two DCA3 drip covers
- Two SIFCA-CPVC for BX3000-18
- Two single-pipe bottom sparging system

**Tank 12-b****780 Gallon Tank (47 x 47 x 70 in. deep) with Maximum 12-in. Freeboard**

- One BX3000-18 with Titanium-tip shaft, 90AFR1 adjustable flow restrictor and two PMCXF20 (20-in. long) Permacore Super Magnum Reusable Filters
- One DCA3 drip cover
- One SIFCA-CPVC for BX3000-18
- One dual-pipe bottom sparging system

**Tank 12-c****1050 Gallon Tank (96 x 30 x 96 in. deep) with Maximum 12-in. Freeboard**

- One BX3000-18 with Titanium-tip shaft, 90AFR1 adjustable flow restrictor and two PMCXF20 (20-in. long) Permacore Super Magnum Reusable Filters
- One DCA3 drip cover
- One SIFCA-CPVC for BX3000-18
- One single-pipe bottom sparging system



4 Demonstration Activities – Plating Quality

Two test phases were conducted at ANAD to determine the effects of the PRD-EL1 on the plating quality. Phase I was conducted from 26–30 August 2002, while Phase II was conducted from 2–6 June 2003. This chapter describes the operation and fluid sampling during the demonstration. Additional information regarding this demonstration can be found in the appendices. A complete set of photographic images taken during the demonstration are shown in Appendix E, and a photocopy of the field notes is contained in Appendix F.

Demonstration Operation

Parts were selected from production lines in ANAD and CCAD. Part numbers, specifications, and associated drawings were collected for reference during testing and analysis. Tables 2 and 3 list parts tested during Phases I and II.

Table 2. Parts tested during Phase I.

Part Number	Common Name	Test Performed			Originating Facility
		Baseline	2-in. PRD	4-in. PRD	
114L2425-1	Piston	x	x		CCAD
145H7359-1	Rod	x		x	CCAD
12304148	Adapter	x	x	x	ANAD
12286191	LP Shaft	x	x	x	ANAD
12284387	Spur Gear	x	x	x	ANAD
Test coupons	Coupons	x	x	x	ANAD

Table 3. Parts tested during Phase II.

Part Number	Common Name	Test Performed			Originating Facility
		Baseline	2-in. PRD	4-in. PRD	
12304148	Adapter	x	x	x	ANAD
12286191	LP Shaft	x	x	x	ANAD
Test coupons	Coupons	x	x	x	ANAD

The system was operated in a batch mode. Parts were lowered into the tank utilizing existing handling systems (Figure 3). Figures 4 and 5 show how the parts were arranged in the tank.

The tank solution was sampled before, during, and after each plating batch. Only one sample of the PRD fluid was taken during the entire Phase I test. During Phase II, however, PRD fluid samples were taken before, during, and after each plating batch along with the tank solution samples. These samples were analyzed to determine background contaminant levels. Parts used during the demonstration were plated per ANAD protocols for a period of 12 hours under normal conditions.

During Phase I, the baseline test was conducted first. After the baseline test was complete, PRD-EL1 liquid was added to the tank at various locations. Several parts were plated with an initial PRD-EL1 depth of 2 in., followed by another plating run with the PRD-EL1 fluid maintained at a depth of 4 in.

During Phase II, the baseline test was conducted last. PRD-EL1 liquid was added to the tank at various locations in preparation for the first plating cycle. Several parts were plated with an initial PRD-EL1 depth of 4-in., followed by another plating run with the PRD-EL1 fluid maintained at a depth of 2 in.



Figure 3. Test parts positioned on handling systems.

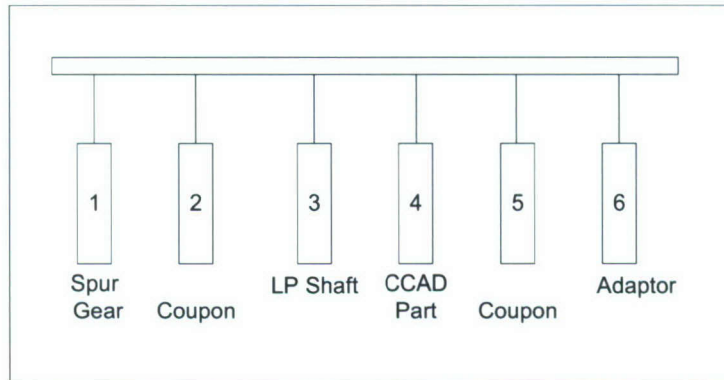


Figure 4. Arrangement of parts within the tank during Phase I.

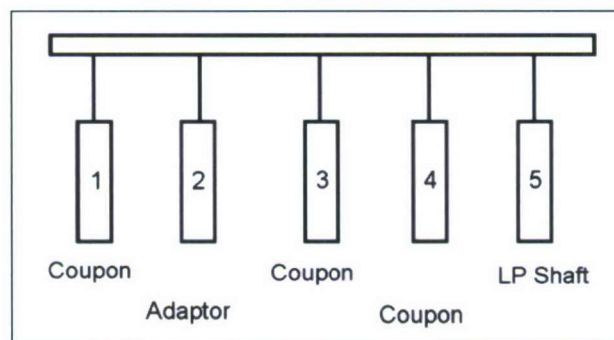


Figure 5. Arrangement of parts within the tank during Phase II.

The liquid depth was measured using a beaker and measuring tape (Figure 6). Depths were recorded at the same temperature to account for expansion of the liquid before each test run and at the completion of the run. Amperage, time, voltage, and temperature were also recorded during testing at selected intervals.

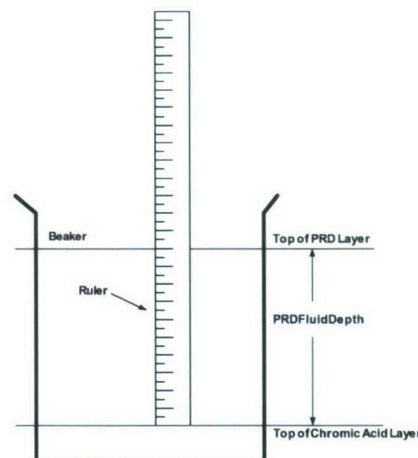


Figure 6. Depth of liquid measured using a beaker and measuring tape.

Fluid Sampling

Phase I

Phase I testing was divided into three different plating cycles: Baseline (no PRD fluid), 2-in. PRD Fluid Level, and 4-in. PRD Fluid Level. Table 4 is a quick summary of the test. All chemical samples were hermetically sealed and labeled for date, time, and sample identification. Notations of the sample collections were also recorded in the logbook (Appendix F).

Baseline Level

Although all relevant parts were plated, only one sample of the actual chromic acid solution was taken to be analyzed as a baseline/control for the rest of the samples.

Table 4. Phase I test summary.

Plating Cycle	Day	Time (hr)	Step in Process	Voltage	Amps	Temperature (°F)	Samples Taken	PRD Fluid Depth (in.)
Baseline	8/26/02	13:10	Reverse Etch	1	210	130	Baseline	
		13:13	Start	3.6	212	133		
		13:43		3.6		130		
		14:15		3.6		133		
		15:30		4.2	218	125		
	8/27/02	01:00	End	4.2	218	125		
2-in. PRD Fluid		10:10	Reverse Etch	1	210	133	A	2
		10:13	Start	3.6	230	132		2
	8/27/02	16:11	Middle	3.9	235	132	B	2
		21:40	End	3.8	236	128	C	2
4-in. PRD Fluid		06:50	PRD Fluid Added	0	0	121	C1	4
		08:00					D	
		09:28	Reverse Etch	1	180	136		
	8/28/02	09:32	Start	4	240	133		
		15:05	Middle	4	250	133	E	3.5
		21:00	End	3.8	250	131	F	3.75

2-in. PRD Fluid Level

Approximately 15 gallons of PRD-EL1 fluid (clear in color) was added to the plating bath, creating a layer roughly 2 in. in depth. Samples of chromic acid solution were taken at various locations within the plating bath using a syringe so as not to contaminate the sample with PRD fluid. Figure 7 shows a representation of how this was accomplished.

During this plating cycle, it was recorded in the logbook that the PRD-EL1 solution had turned to a dark yellow (almost mustard) color. Five minutes after the end of this plating cycle, however, the color of the PRD-EL1 fluid appeared to become clear again.

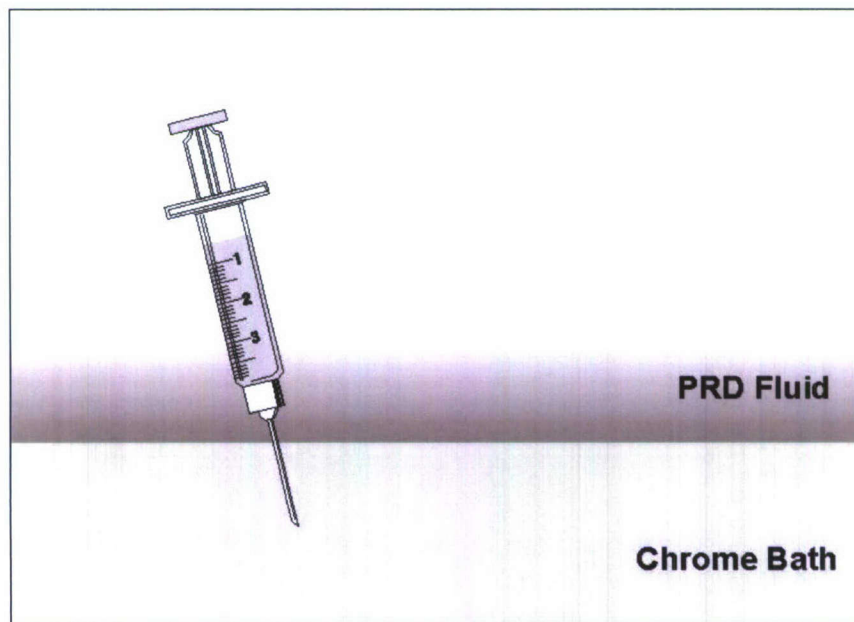


Figure 7. A representation of the sampling.

4-in. PRD Fluid Level

The PRD-EL1 fluid appeared to gain a brown-reddish pallor overnight. Because of the unusual color, a sample of the PRD-EL1 solution was taken. The sample was hermetically sealed, labeled as a special sample (C1), and date and time were recorded. Following this step, the extra PRD-EL1 fluid (approximately 15 gallons) was added to the existing tank to bring the PRD-EL1 blanket up to a 4-in. depth.

After an hour of plating, it was recorded in the logbook that the PRD-EL1 solution had again turned a dark yellow (almost mustard) color. Later in the day, however, it was noted that the bubbles breaking through the fluid were different colors (Figure 8). Bubbles closer to the pump side of the tank turned clear before bursting; however, bubbles bursting farthest from the pump remained black, suggesting that chromium emissions were getting through.

During this time, the amperages at each point on the cathode bar and on one anode bar were measured. Figure 9 shows the results of the measurements. At the completion of the demonstration, all parts and samples were packaged for shipment to Benét Laboratories for analysis.

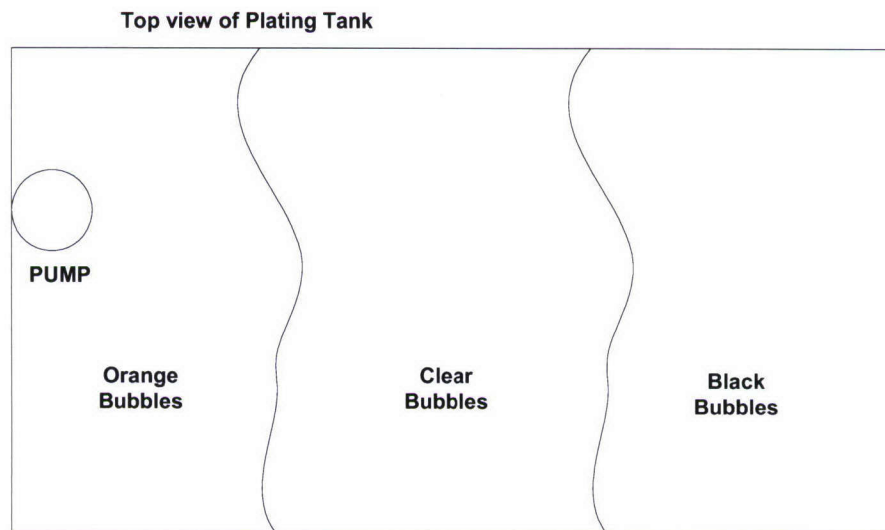


Figure 8. Bubbles breaking through the fluid.

Current Measurements at 4" PRD Level
After Cleaning Cathode Bar

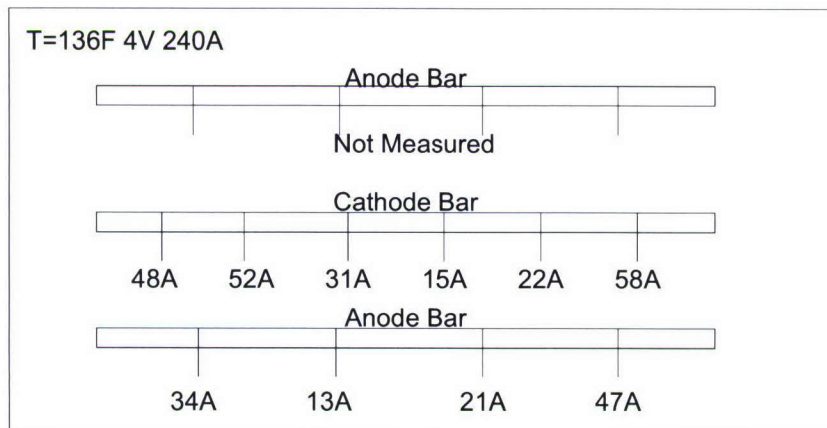


Figure 9. Phase I current measurements.

Phase II

Phase II testing was divided into three different plating cycles: 4-in. PRD Fluid Level, 2-in. PRD Fluid Level, and Baseline (no PRD fluid). Table 5 is a quick summary of the Phase II test.

Table 5. Phase II test summary.

Day	Time (hr)	Step in Process	Voltage	Amps	Temperature (°F)	Samples Taken	PRD Fluid Depth (in.)	Notes
6/3/03	0825	Sample	-	-	110	Xa, A, B	4	
	1030	Reverse Etch	-	300	131		4	
	1032	Start Plate	4.2	200 +/- 10	131		4	
	1627	Sample	4.7	298 +/-5	131	C, D	3.75	
	2215	Sample	4.6	307 +/-5	132	E, F	3.75	
	2237	End Plate						
6/4/03	0740	Sample	-	-	130	G, H	2.25	Amperage not stable
	0828	Reverse Etch	-	250	130		2.25	
	0830	Start Plate	4.1	285 +/-10	129		2.25	
	1415	Sample	4.2	270 +/-10	129	I, J	2.25	
	1930	Sample	1.2	157	129	K, L	2.25	
	2000	End Plate						
6/5/03	0800	Sample	-	-	120	M	0	
	0830	Reverse Etch		300	121		0	
	0833	Start Plate	4.2	245 +/-5	121		0	
	1400	Sample	4.7	296	125	N	0	
	1930	Sample	4.6	305	130	O	0	
	2000	End Plate					0	

All chemical samples were hermetically sealed and labeled for date, time, and sample identification. Notations of the sample collections were also recorded in the log-book. During this phase, amperage measurements were taken at each of the parts positions. These measurements were taken at various times during each plating cycle by utilizing an ohmmeter provided by ANAD. Table 6 summarizes the results of current measurements.

Table 6. Phase II current measurements summary.

Condition	Time	Cathode Current Measurements (Amps)						Comments
		Position					Total	
		Coupon	Adaptor	Coupon	Coupon	LP Shaft	From Rectifier	
4-in.	1047	70	49	65	40	10	200 +/- 10	Jumping Jumping Swinging wildly Stable Stable
	1627	84	58	64	58	30	298 +/- 5	
	2200	81	57	67	61	34	307 +/- 5	
2-in.	0840	61	39	54	54	11	235 +/- 10	
	1400	61	22	29	69	15	270 +/- 10	
	1956	35	25	28	35	9	157	
0-in.	0840	68	38	58	68	17	245 +/- 5	
	1415	70	51	67	89	21	296	
	1930	64	50	68	90	23	305	

4-in. PRD Fluid Level

The PRD-EL1 fluid was added to the tank on 2 June 2003. On the morning of 3 June, what appeared to be a brown-reddish gelatinous substance had formed in the tank overnight. A special sample of this substance was taken and labeled Xa. Although a picture was taken of this substance in tank 12-b, the picture of the same type of substance in tank 12-a (Figure 10) was much clearer.

Samples of chromic acid solution and PRD fluid were taken throughout the plating cycle at various locations within the plating bath. The samples of chromic acid solution were taken using a syringe so as not to contaminate the sample with PRD fluid (Figure 7).

Measurements of the total amperage and the individual amperage at each anode were taken at roughly the same time as the samples.

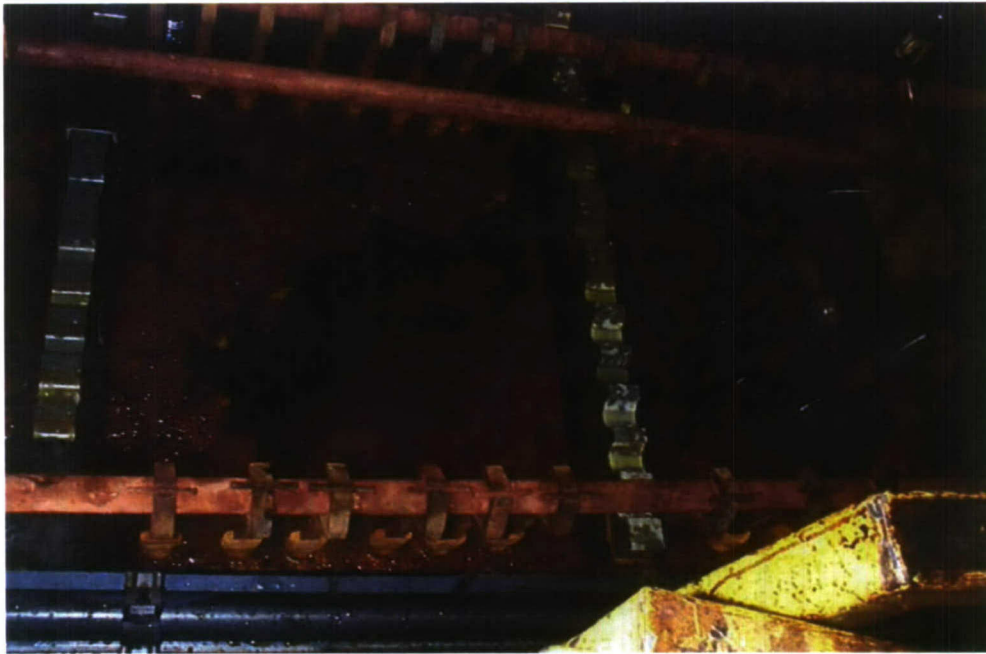


Figure 10. Brown-reddish gelatinous substance in 12-a.

2-in. PRD Fluid Level

Approximately 15 gallons of PRD-EL1 fluid were removed from the plating bath, creating a layer roughly 2-in. in depth. Plating and sampling were conducted in the same manner as in the 4-in. plating cycle. However, the amperage during this plating cycle was not steady and actually swung from 50 amps to more than 290 amps throughout most of the latter half of the test. Although many theories as to the cause of this instability were discussed in the field, no definitive answers were found.

Baseline Level

Morning, afternoon, and evening samples of chromic acid solution were taken as a baseline/control for the rest of the samples to be analyzed against. At the completion of the Phase II tests, all parts and samples were packaged for shipment to Benét Laboratories for analysis.

5 Chemical Analysis

The chemical laboratory at Benét Laboratories analyzed all chromium solution and PRD-EL1 fluid samples from Phases I and II. The purpose of the analyses was to determine if the plating solution was chemically affected by the presence of the PRD-EL1 fluid.

The samples from Phase I were analyzed for the four following elements: chromic acid, sulfuric acid, iron, and trivalent chromium. Table 7 shows Phase I samples.

The chromium solution samples from Phase II were analyzed for chromic acid and sulfuric acid. The PRD fluid samples from Phase II were analyzed for iron and total chromium. Table 8 shows Phase II samples.

Table 7. Phase I samples.

Sample	Description
Baseline	Initial sample taken from normal plating cycle (1 st day)
A	Morning sample taken from 2-in. PRD plating cycle (2 nd day)
B	Afternoon sample taken from 2-in. PRD plating cycle (2 nd day)
C	Evening sample taken from 2-in. PRD plating cycle (2 nd day)
C1	* Special sample taken of PRD due to unusual color (3 rd day)
D	Morning sample taken from 4-in. PRD plating cycle (3 rd day)
E	Afternoon sample taken from 4-in. PRD plating cycle (3 rd day)
F	Evening sample taken from 4-in. PRD plating cycle (3 rd day)

Table 8. Phase II samples.

Sample	Description
Xa	Sample of odd material taken before first plating cycle (1 st day)
A	Morning sample of chromium taken from 4-in. PRD plating cycle (1st day)
B	Morning sample of PRD taken from 4-in. PRD plating cycle (1st day)
C	Afternoon sample of chromium taken from 4-in. PRD plating cycle (1st day)
D	Afternoon sample of PRD taken from 4-in. PRD plating cycle (1st day)
E	Evening sample of chromium taken from 4-in. PRD plating cycle (1st day)
F	Evening sample of PRD taken from 4-in. PRD plating cycle (1st day)
G	Morning sample of chromium taken from 2-in. PRD plating cycle (2 nd day)
H	Morning sample of PRD taken from 2-in. PRD plating cycle (2 nd day)
I	Afternoon sample of chromium taken from 2-in. PRD plating cycle (2 nd day)
J	Afternoon sample of PRD taken from 2-in. PRD plating cycle (2 nd day)

Sample	Description
K	Evening sample of chromium taken from 2-in. PRD plating cycle (2 nd day)
L	Evening sample of PRD taken from 2-in. PRD plating cycle (2 nd day)
M	Morning sample of chromium taken from baseline plating cycle (3rd day)
N	Afternoon sample of chromium taken from baseline plating cycle (3rd day)
O	Evening sample of chromium taken from baseline plating cycle (3rd day)

Chromic Acid (CrO₃)

The analysis was performed using a titration of ferrous ammonium sulfate and sulfuric acid. To ensure the quality of the results, a known standard was titrated at the same time. Each sample was analyzed twice and an average was computed. The results are shown in Figures 11 (Phase I) and 12 (Phase II).

ANAD has specified that CrO₃ within plating tanks should be maintained at levels between 164 to 493 g/l. Through years of experience with the plating process, WVA has determined that optimum plating specifications for chromic acid are achieved when CrO₃ is maintained at a minimum of 250 g/l \pm 10 g/l.

As can be seen in Figures 11 and 12, the samples taken during this demonstration passed the acceptable limits imposed by ANAD with the exception of sample M (Figure 12) from Phase II.

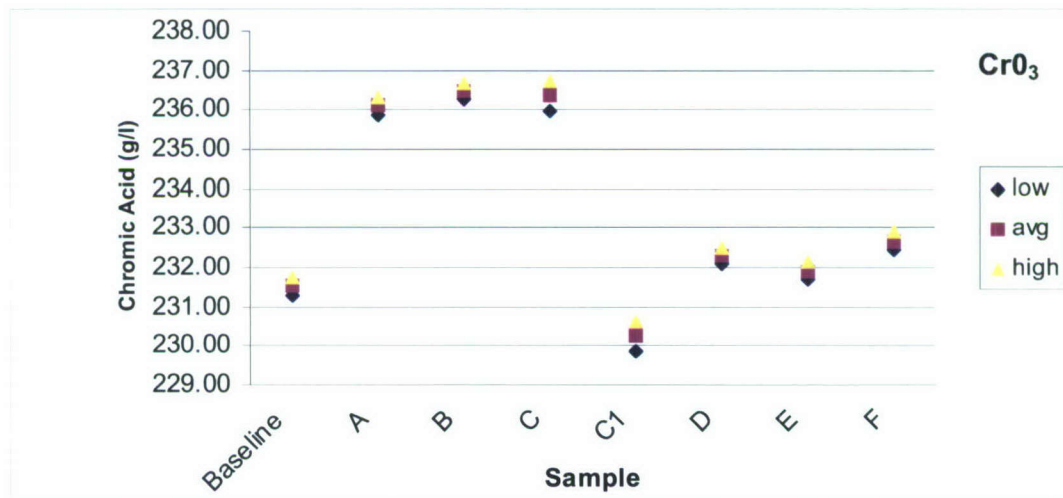


Figure 11. Phase I chromic acid results.

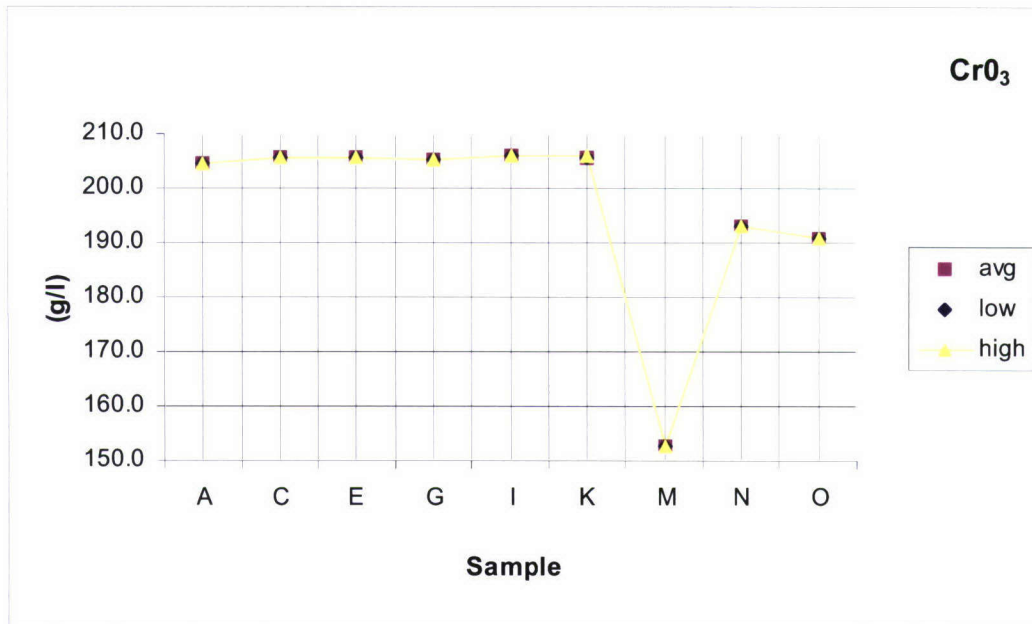


Figure 12. Phase II chromic acid results.

Sulfuric Acid (H₂SO₄)

Analysis was performed using a DX-120 Ion Chromatograph (Dionex, Sunnyvale, CA). For quality assurance purposes, the instrument was calibrated before each run, and a known standard was run along with the samples. Each sample was analyzed twice, and an average was computed. Figures 13 and 14 show the results for Phases I and II, respectively.

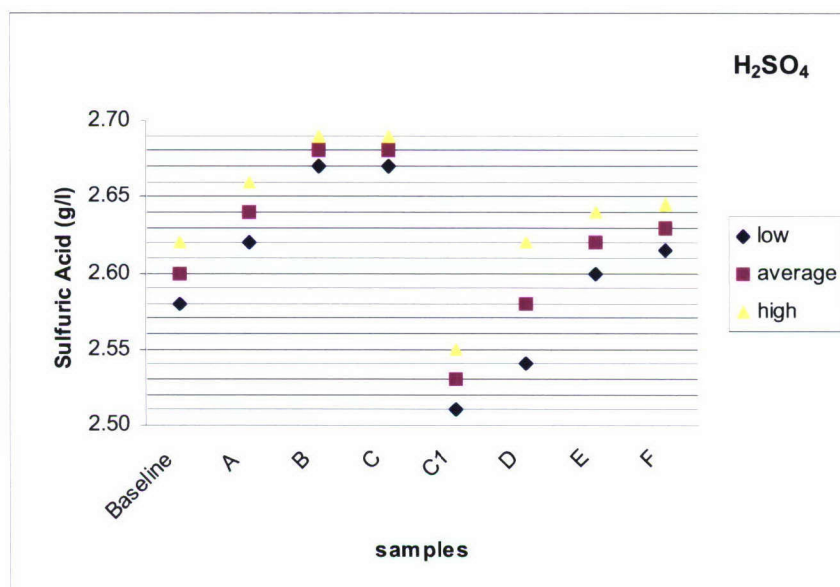


Figure 13. Phase I sulfuric acid results.

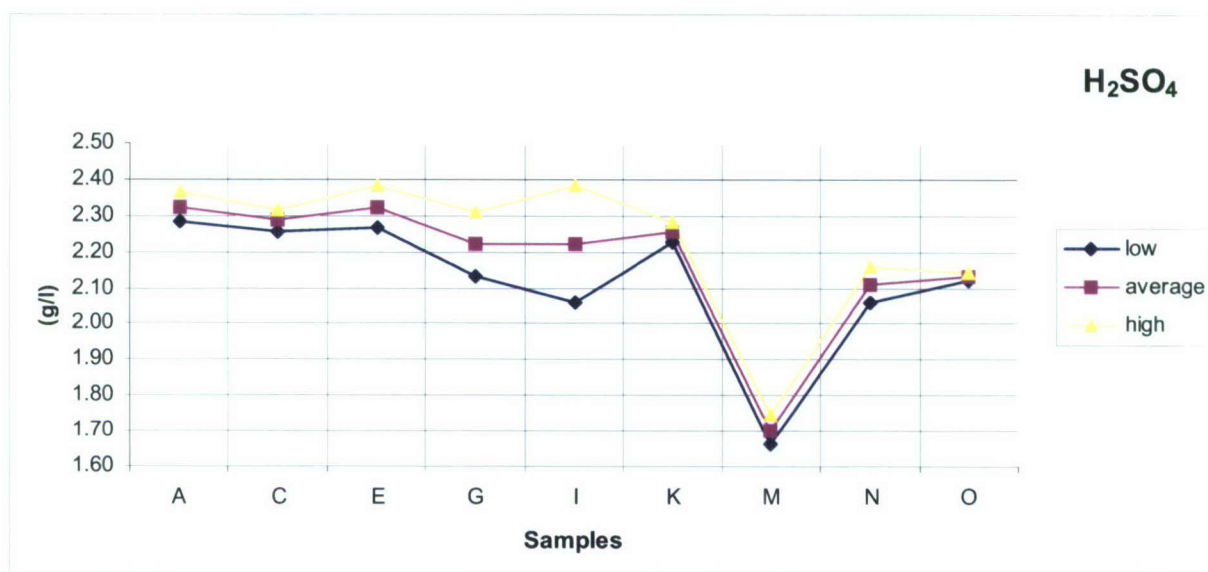


Figure 14. Phase II sulfuric acid results.

ANAD has specified that H_2SO_4 within plating tanks should be maintained at levels between 1.64 to 4.93 g/l (essentially a 100 to 1 ratio when compared to CrO_3). This specification was met by all samples. WVA has determined that optimum plating specifications for chromic acid are achieved when CrO_3 is maintained at a minimum of $2.5 \text{ g/l} \pm 0.1 \text{ g/l}$. When compared with the WVA specifications for CrO_3 , however, the WVA also uses a 100 to 1 ratio.

Iron (Fe)

Analysis was performed using Inductive Coupled Plasma Optical Emission Spectrometry. An Optima 3100XL Spectrometer (PerkinElmer, Wellesley, MA) was used. Once again, for quality assurance purposes, the instrument was calibrated before each run and a known standard was run with the samples. Each sample was analyzed twice, and an average was computed. Figures 15 and 16 show the results for Phases I and II, respectively.

ANAD has specified that Fe within plating tanks should be maintained at levels below 7.5 g/l. WVA has also determined that optimum plating specifications for chromic acid are achieved when Fe is maintained at a level below 7.5 g/l. Figures 15 and 16 show that the chromium samples from Phase I and most of the PRD samples from Phases I and II meet this specification. However, samples Xa and J from Phase II indicate large amounts of iron suspended in the PRD fluid.

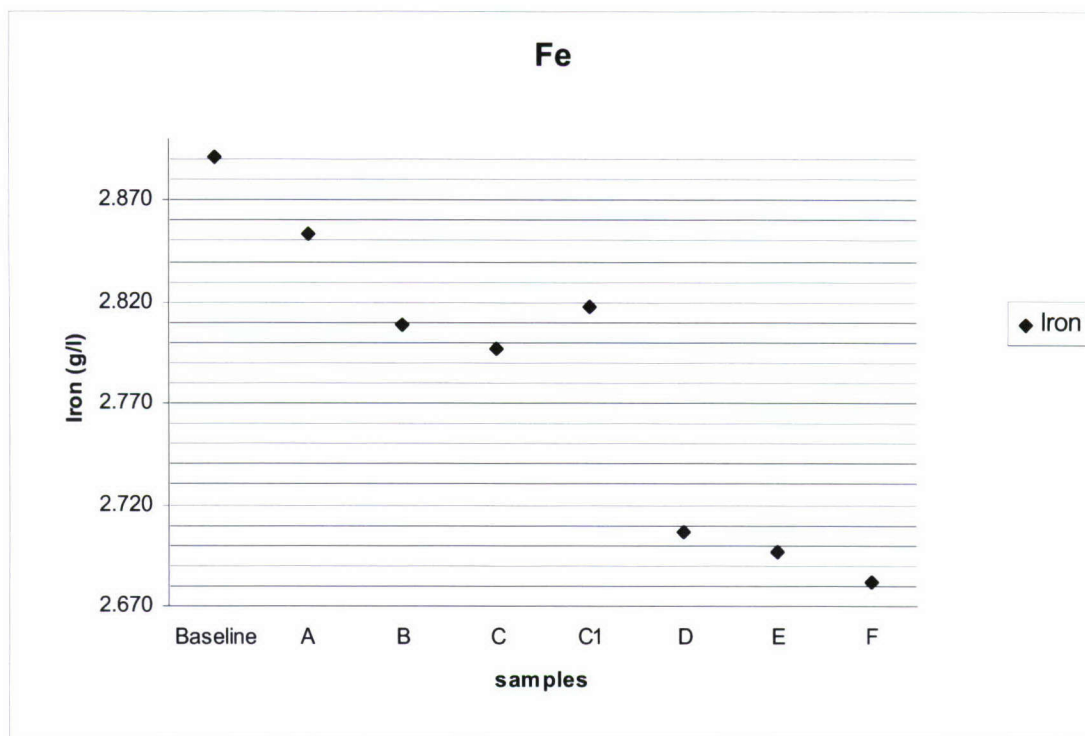


Figure 15. Phase I iron results.

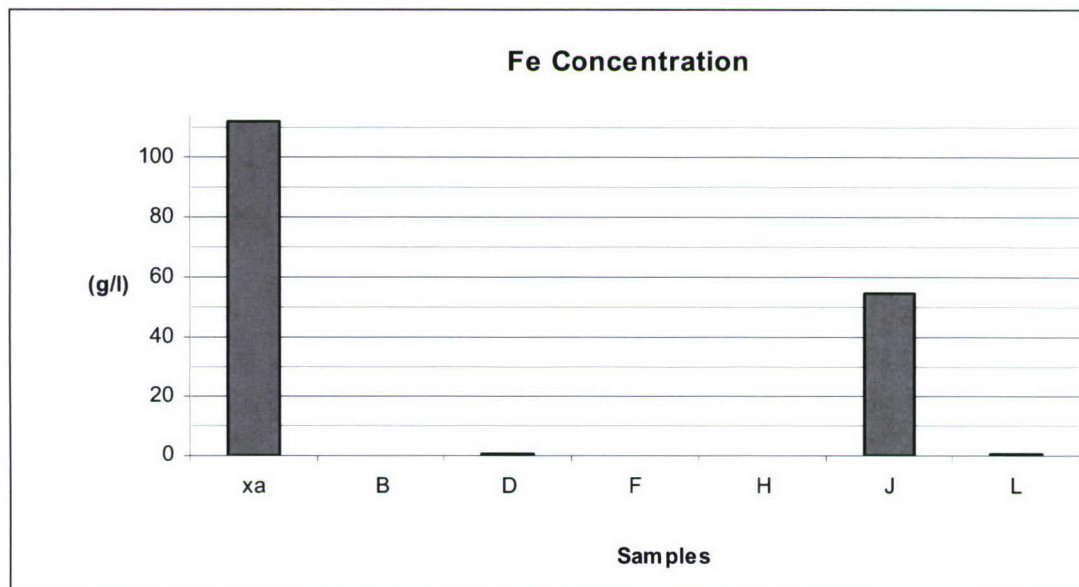


Figure 16. Phase II iron results.

Trivalent Chromium

Phase I

Analysis for Phase I was performed using a PerkinElmer Lambda 40 Ultraviolet/Visible (UV/Vis) Spectrometer. The instrument was calibrated before use, and a known standard was run with the samples. Each sample was analyzed twice, and an average was computed. Figure 17 shows the results.

ANAD has specified that Trivalent Chromium [Cr (III)] within plating tanks should be maintained at levels below 7.5 g/l. WVA has also determined that optimum plating specifications for chromic acid are achieved when Cr (III) is maintained at a minimum of 7.5 g/l. As shown in Figure 17, the chromium solution samples taken during this phase did not show excessive amounts of trivalent chromium. The special sample taken (C1) has a higher level of trivalent chromium than the other samples, but still meets the specification.

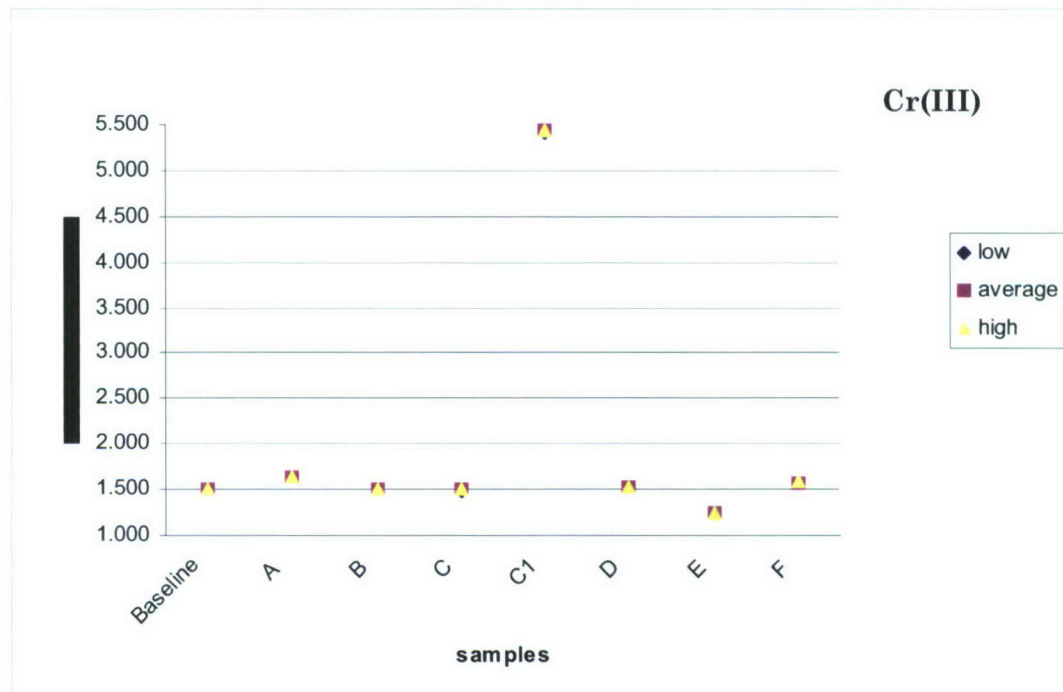


Figure 17. Phase I trivalent chromium results.

Phase II

Only the PRD fluid samples were analyzed for the presence of trivalent chromium during the Phase II chemical analyses. In contrast to Phase I, total chromium was measured instead of just trivalent chromium using an ICP Emission Spectrometer. The instrument was calibrated before use and a known standard was run with the samples. Each sample was extracted by diluting it with nitric acid and then heated for 24 hours at approximately 75 °C. The samples were then placed in a separatory funnel to cool, and the chromium was extracted. Figure 18 shows the results of the total chromium extraction.

With the possible exception of sample Xa, the PRD samples taken during Phase II did not show excessive amounts of trivalent chromium. The special sample (Xa – brown-reddish gelatinous substance found in the electroplating tank the morning after PRD fluid was added to the tank) suggests an interesting reaction may have occurred between the plating solution and the PRD fluid. The sampled material broke up easily and appeared to be an emulsion; thus, the reaction may result in an emulsion that floats to the surface of the PRD fluid.

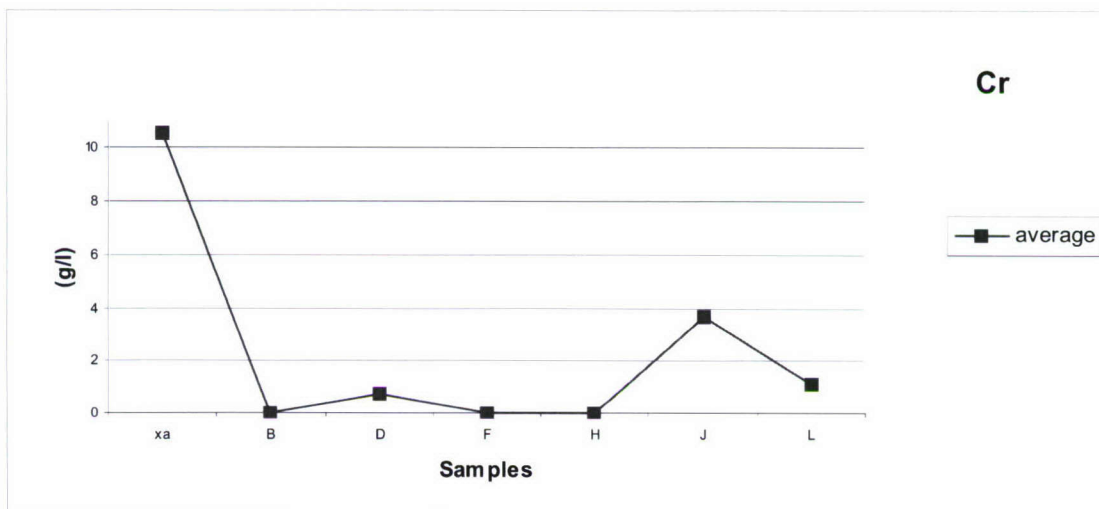


Figure 18. Phase II total chromium in PRD fluid results.

Summary

Tables 9 and 10 summarize the chemical analysis results from Phases I and II. Appendices A and B show the tabulated form of the chemical analysis results for Phases I and II, respectively.

Table 9. Results summary of Phase I chemical analysis.

Sample	CrO ₃ (g/l) (average)	H ₂ SO ₄ (g/l) (average)	Fe (g/l) (average)	Cr III(g/l) (average)
Baseline	231.53	2.60	2.891	1.502
A	236.10	2.64	2.853	1.640
B	236.48	2.68	2.809	1.507
C	236.35	2.68	2.797	1.499
C1	230.26	2.53	2.818	5.427
D	232.30	2.58	2.707	1.525
E	231.91	2.62	2.697	1.247
F	232.68	2.63	2.682	1.578

Table 10. Results summary of Phase II chemical analysis.

Sample	CrO ₃ (g/l) (average)	H ₂ SO ₄ (g/l) (average)	Fe (g/l) (average)	Cr (g/l) (average)
Xa			112	10.56
A	204.54	2.32		
B			0.0002	0.018
C	205.68	2.29		
D			0.4	0.740
E	205.55	2.32		
F			0.04	0.010
G	205.04	2.22		
H			0.002	0.02
I	205.81	2.22		
J			54.4	3.684
K	205.68	2.25		
L			0.4	1.088
M	152.58	1.70		
N	193.10	2.11		
O	190.69	2.13		

The Phase I results indicate that all of the plating solution samples were consistent with the baseline samples and met optimum plating specifications imposed by ANAD for chromic acid, sulfuric acid, iron, and trivalent chromium. Phase II plating solution samples also met the ANAD specifications for chromic acid and sulfuric acid with the exception of sample M, which was a baseline sample. These results suggest that the plating solution was not adversely affected by the presence of the PRD-EL1 fluid.

One PRD-EL1 fluid sample (C1) from Phase I and seven PRD-EL1 fluid samples from Phase II were analyzed. Sample C1 was shown to contain a higher level of trivalent chromium than the plating solution samples. Surprisingly, C1 contained similar amounts of chromic acid, sulfuric acid, and iron as contained in the plating

solution samples. Because of these concentrations, it is most likely that C1 was actually a sample of the plating solution near the fluid interface rather than the PRD-EL1 fluid. Sample Xa contained higher levels of chromium than did the other PRD-EL1 fluid sample, but this sample and the others from Phase II did not indicate a total chromium concentration that would be expected in C1 or the plating solution samples. In addition, samples Xa and J contained much more iron than the other PRD-EL1 fluid samples and the plating solution samples from Phase I. These data and the observations noted in the previous chapter indicate that the PRD-EL1 fluid would on occasion either become difficult to distinguish from the chromic acid solution or undergo an undetermined reaction that changed the appearance and chemical makeup of the fluid.

6 Material Analysis

Phase I

The parts were individually photographed and catalogued by the Benét Materials Laboratory. A short scope of work was identified to determine the type of testing that would be most relevant in ascertaining if the parts would meet Army specifications. Each component and coupon was subjected to the following American Society for Testing and Materials (ASTM) and Society of Automotive Engineers tests/evaluations:

- a. Thickness Testing in accordance with ASTM B487
- b. Adhesion Testing in accordance with ASTM B571 (para. 8)
- c. Quality Testing in accordance with AMS 2406K (para. 3.5)
- d. Porosity Testing in accordance with AMS-QQ-C-320
- e. Hardness Testing in accordance with ASTM B578.

Because each part was plated for 12 hours regardless of its specified plating time, hydrogen testing (in accordance with ASTM F 519) was not performed. Table 11 summarizes the results obtained.

The results show that 3 of the 13 components, and 2 of the 3 coupons failed at least one test. One of the component failures was actually a component subjected to the conventional electroplating process (i.e., no PRD-EL1 fluid blanket). Additionally, microhardness and thickness of the plating were noticeably decreased in the components PRD-processed (114L2425-1 and 145H77359-1 series) when compared to their conventionally plated counterpart components. This decrease was more pronounced in component 114L2425-1 2-in. PRD.

The entire report received from the Benét Materials Laboratory for Phase I can be found in Appendix C. Appendix G contains a discussion of the results presented on Phase I by the process inventor and offers explanations for some of the variations in plating quality. During the demonstration, ANAD conducted the plating operations in a manner consistent with normal operations. The only difference was the pres-

ence of the PRD-EL1 fluid. Although the observations detailed in Appendix G may explain some of the failures and variation in quality, it is highly unlikely that special operational changes can be made for the purpose of using PRD-EL1 as the chromium emission control method.

Table 11. Summary of Phase I materials analysis.

Part Identification	Thickness (in.)	Adhesion (Pass/Fail)	Quality (Pass/Fail)	Porosity (Pass/Fail)	Microhardness (HV)*
114L2425-1 Baseline	0.0113	Pass	Pass	Pass	Pass (906.90)
114L2425-1 2-in. PRD	0.0040	Pass	Pass	Pass	Pass (824.20)
145H7359-1 Baseline	0.0137	Fail	Pass	Pass	Pass (921.33)
145H7359-1 4-in. PRD	0.0126	Pass	Pass	Pass	Pass (885.87)
12304148 Baseline	0.0074	Pass	Pass	Pass	Pass (855.33)
12304148 2-in. PRD	0.0070	Pass	Fail	Pass	Pass (858.60)
12304148 4-in. PRD	0.0094	Pass	Pass	Pass	Pass (858.60)
12286191 Baseline	0.0232	Pass	Pass	Pass	Pass (877.07)
12286191 2-in. PRD	0.0131	Pass	Pass	Pass	Pass (885.47)
12286191 4-in. PRD	0.0129	Pass	Pass	Pass	Pass (894.40)
12284387 Baseline	0.0149	Pass	Pass	Pass	Pass (885.33)
12284387 2-in. PRD	0.0097	Pass	Pass	Pass	Pass (856.07)
12284387 4-in. PRD	0.0183	Pass	Fail	Pass	Pass (835.07)
Baseline Coupons	0.0038	Pass	Pass	Pass	Pass (844.87)
2-in. PRD Coupons	0.0031	Fail	Pass	Pass	Pass (837.40)
4-in. PRD Coupons	0.0060	Fail	Pass	Pass	Pass (893.00)
HV – A unit of hardness based on the Vickers Method.					

Phase II

Testing was conducted in accordance with AMS 2406K (“Plating, Chromium Hard Deposit,” para. 4.2.3) and AMS-QQ-C320 (“Chromium Plating”, para. 3.4.2 and 4.5.4). Table 12 identifies the supplied components and coupons.

Table 12. Supplied components and coupons.

Components/Coupons
12304148 Baseline
12304148 2-in. PRD
12304148 4-in. PRD
12286191 Baseline
12286191 2-in. PRD
12286191 4-in. PRD
Baseline coupons
2-in. PRD coupons
4-in. PRD coupons

All components and coupons were produced at ANAD. The components/coupons were subjected to three different chromium plating conditions or lots: baseline (conventional) plating, 2-in. PRD fluid plating, and 4-in. PRD fluid plating. All components were plated for 12 hours duration. As with Phase I, each component and coupon was subjected to the following tests/evaluations:

- a. Thickness Testing in accordance with ASTM B487
- b. Adhesion Testing in accordance with ASTM B571 (para. 8)
- c. Quality Testing in accordance with AMS 2406K (para. 3.5)
- d. Porosity Testing in accordance with AMS-QQ-C-320
- e. Hardness Testing in accordance with ASTM B578.

Results/Discussion

Thickness

Chromium plating thickness was measured by examining a metallographic cross-section of the component/coupon in accordance with ASTM B487. Multiple cross-sections were obtained from each component/coupon. These cross-sections were mounted, polished, and etched with a 2 percent nital solution.

Table 13 shows the thickness of the chromium plating for each test piece. Measurements were taken at multiple points on each sample and averaged. Plating thicknesses on components and coupons have no specific requirements, so the values are reported mainly for comparing the varied plating processes.

Table 13. Chromium plating thickness.

Part Identification	Plating Thickness (in.)
12304148 Baseline	0.006
12304148 2-in.	0.003
12304148 4-in.	0.006
12286191 Baseline	0.017
12286191 2-in.	0.013
12286191 4-in.	0.013
Baseline Coupon	0.002 see note
2-in. PRD Coupon	0.001 see note
4-in. PRD Coupon	0.001 see note

Note: The average thickness for 2-in. and 4-in. PRD was below the minimum recommended thickness requirement of AMS 2406K (0.002 in.). Additionally, all three coupon sets had rough substrate surface finishes compared with the actual components. This rougher surface appeared to deleteriously affect the plating process and resulting plating.

Figure 19 shows an example of a cross section used for thickness measurements. Figure 20 is an example cross section showing thickness and substrate finish.

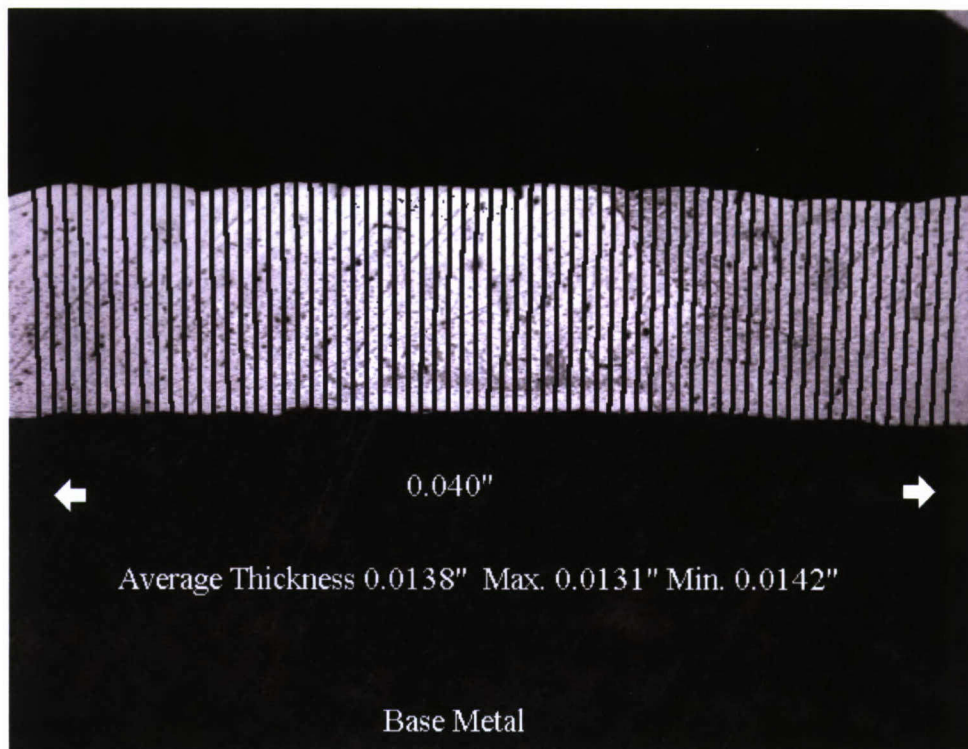


Figure 19. Typical view of chromium plating sample used for thickness measurement.

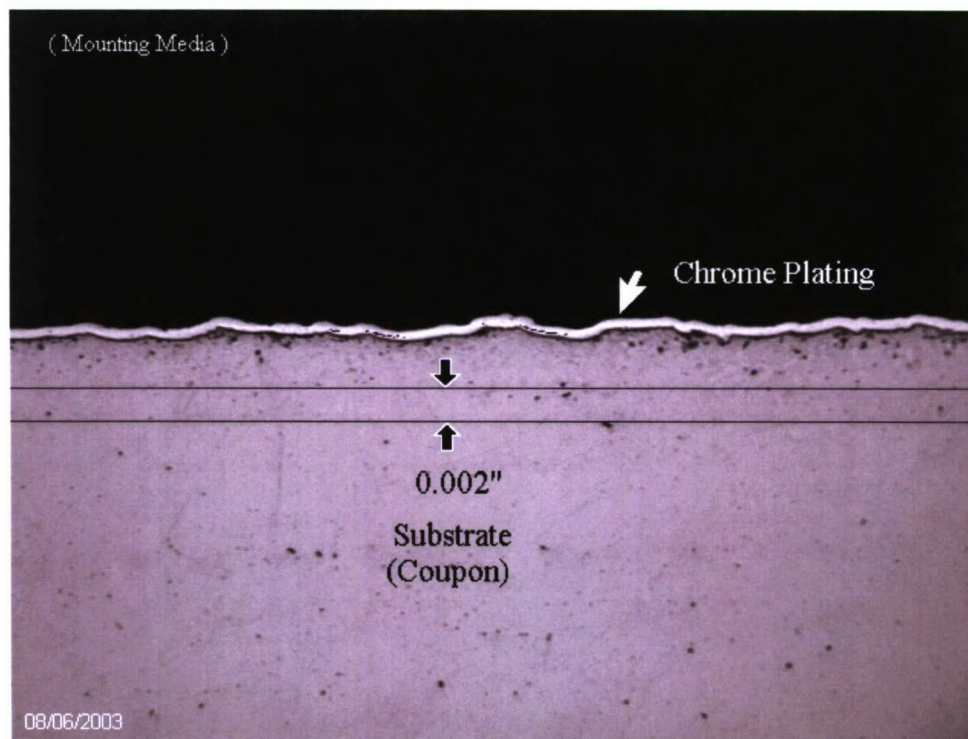


Figure 20. Baseline Coupon with thin plating and rough substrate surface finish.

Adhesion

Adhesion testing was conducted in accordance with ASTM B571 (para.8). The Grind Saw Test was performed on eight areas for each specimen, and the areas were then examined at 5X magnification. Table 14 shows results from this test.

Table 14. Results from the Grind Saw Test.

Part Identification	Adhesion (Pass/Fail)
12304148 Baseline	Fail
12304148 2-in. PRD	Fail
12304148 4-in. PRD	Fail
12286191 Baseline	Pass
12286191 2-in. PRD	Pass
12286191 4-in. PRD	Pass
Baseline Coupons	Fail
2-in. PRD Coupons	Fail
4-in. PRD Coupons	Fail

The ASTM specification states that lifting or peeling of the coating is evidence of unsatisfactory adhesion. Figures 21–24 show some failed samples.

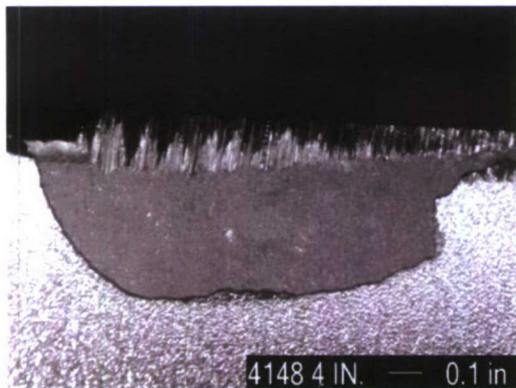


Figure 21. Failed sample (4148 4 in).



Figure 22. Failed sample (4148 Baseline).

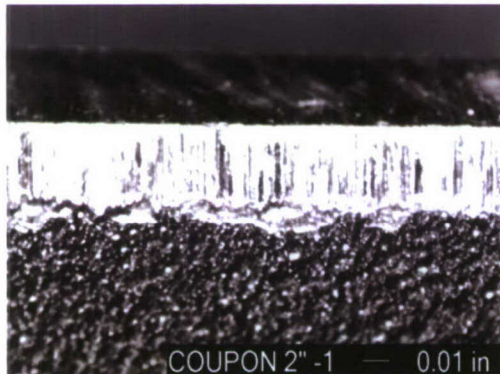


Figure 23. Failed sample (Coupon 2 in).



Figure 24. Failed sample (Coupon Baseline).

Quality

Quality testing was performed on each piece (components, coupons) by visual inspection in accordance with AMS 2406K (para. 3.5). The specification requires that the plating be firmly bonded to the base metal, while maintaining a smooth and uniform appearance. Pinholes and other small imperfections that resulted from the plating not filling in defects on the surface metal are considered acceptable. A magnification of 5X was used to inspect the pieces. Table 15 shows the results.

Table 15. Quality testing results by visual inspection.

Part Identification	Quality (Pass/Fail)
12304148 Baseline	Pass
12304148 2-in. PRD	Pass
12304148 4-in. PRD	Fail
12286191 Baseline	Fail
12286191 2-in. PRD	Pass
12286191 4-in. PRD	Pass
Baseline Coupons	Pass
2-in. PRD Coupons	Pass
4-in. PRD Coupons	Pass

Coupon 12304148 4-in. has several small surface pits (not through to base metal) and 12286191 Baseline has visible pimples, hairline surface cracks (not through to base metal), and a generally rough texture.

Porosity

Porosity testing was conducted in accordance with AMS-QQ-C-320. A ferroxyl test was completed by soaking filter paper in ferroxyl solution and heating to 180–200 °F. The paper was then placed over the entire plated area on each piece for 10 minutes. Blue markings occurred on the filter paper as a result of the reaction of the base metal with the solution. These markings are indicative of porosity. Table 16 shows test results.

Table 16. Results of porosity testing conducted in accordance with AMS-QQ-C-320.

Part Identification	Porosity (Pass/Fail)
12304148 Baseline	Pass
12304148 2-in. PRD	Pass
12304148 4-in. PRD	Pass
12286191 Baseline	Pass
12286191 2-in. PRD	Pass
12286191 4-in. PRD	Pass
Baseline Coupons	Pass
2-in. PRD Coupons	Fail
4-in. PRD Coupons	Fail

Hardness

Hardness testing was completed in accordance with ASTM B578. A Vicker's indenter with a 100-gram load was used. The minimum acceptable hardness as per the standard was 700 HV. The average hardness for all samples was above the 700 HV minimum (Table 17). Additionally, no individual hardness readings were below 700 HV.

Table 17. Average hardness for all samples.

Part Identification	Avg. Microhardness (HV)
1230418- Baseline	949
12304148- 2-in.	933
12304148- 4-in.	940
12286191- Baseline	929
12286191- 2-in.	932
12286191- 4-in.	947
Baseline Coupon	876
2-in. PRD Coupon	*
4-in. PRD Coupon	865

* The hardness of the plating on Coupon 2-in. PRD was too thin to measure at the required HV₁₀₀ load.

Summary

Table 18 summarizes results from Phase II.

Table 18. Summary of Phase II results.

Part Identification		Thickness (in.)	Adhesion (Pass/Fail)	Quality (Pass/Fail)	Porosity (Pass/Fail)	Microhardness (HV)
12304148	Baseline	0.006	Fail	Pass	Pass	Pass (949.0)
	2-in. PRD	0.003	Fail	Pass	Pass	Pass (933.0)
	4-in. PRD	0.006	Fail	Fail	Pass	Pass (940.0)
12286191	Baseline	0.017	Pass	Fail	Pass	Pass (929.0)
	2-in. PRD	0.013	Pass	Pass	Pass	Pass (932.0)
	4-in. PRD	0.013	Pass	Pass	Pass	Pass (947.0)
Coupons	Baseline	0.002	Fail	Pass	Pass	Pass (876.0)
	2-in. PRD	0.001	Fail	Pass	Fail	No result - too thin
	4-in. PRD	0.001	Fail	Pass	Fail	Pass (865.0)
HV – A unit of hardness based on the Vickers Method.						

Table 18 shows that four of the six components and the three coupons failed at least one test. All components and coupons subjected to the conventional electroplating process failed at least one of the tests. Appendix D contains the Phase II Benét Materials Laboratory report.

7 Air Sampling Results

The U.S. Army Center for Health Promotion and Preventive Medicine (CHPPM) conducted air sampling tests during Phase II. The sampling included both stack analysis for process emissions and indoor air sampling for worker exposure. The complete results submitted by CHPPM are shown in Appendix I (Volume 2 of this report). Those results are summarized in this chapter.

Stack Sampling

ANAD has two chromium electroplating lines, and each has a separate air handling system. The stack testing occurred on plating line 2 because that was the line with the Zero Emissions System installed. During the stack sampling, the demisters were removed from the exhaust stack so that the process effluent could be tested without removing the aerosols. The ductwork for plating line 2 was replaced prior to the Phase II tests so that there was minimal residual chromic acid in the ducts at the beginning of the tests. Tank 12-a (also containing the appropriate level of PRD-fluid) was put into operation to substantially increase the amperage (by plating a sacrificial part). Three separate sampling runs were conducted for each plating test, and the average of the three runs was used to evaluate whether the process meets the effluent standard (0.015 mg/dscm, Chromium Electroplating NESHAP). During Run 1, when the PRD fluid level in the tanks was 4-in., it was discovered that a tank on Line 1 was in operation. This additional processing had the effect of increasing indoor air concentrations and potentially the stack concentration. The tank was covered with plastic for the remainder of the tests. The data from Run 1 have been eliminated from the average calculations for both the stack emissions and indoor air samples. Table 19 summarizes the stack emissions results.

Table 19. Stack emissions summary results.

Test Series	4-in. PRD-EF1	2-in. PRD-EL1	No PRD-EL1
Amperage			
Tank 12-a	1,100	1,100	1,100
Tank 12-b	300	300	300
Total Amperage	1,400	1,400	1,400
Stack Emission Data			
Total Cr mg/dscm	0.008	0.008	0.014

The results show that use of the PRD-EL1 fluid blanket reduced emissions compared with no PRD-EL1 fluid and with a level below the standard. Surprisingly, the standard was also achieved by the baseline tests in which no PRD-EL1 was used. It was noted during the baseline tests (no PRD-EL1) that a slight but visible amount of PRD fluid remained at the surface of the tank. These baseline results are suspect because the emission creation process depends strongly on the conditions of the plating surface. Therefore, no conclusions can be made about the level of reduction, and it is highly likely that the baseline result is low.

Indoor Air

Indoor air samples were collected inside the plating facility using Total Suspended Particulate (TSP) samplers. These samplers are designed to collect all airborne particles and low vapor pressure liquid particles (aerosols) from approximately 0.8 μm to greater than 100 μm . Details of the collection system and sampling procedures are documented in Appendix I. One sampler was placed to the west of Plating Line 2, 6 ft away from tank 12-b. The other sampler was placed to the east of Plating Line 2, 12 ft away from tank 12-b. The west sampler was in between Plating Lines 1 and 2, and the east sampler was on the opposite side of Plating Line 2. Thus, the west sampler was subject to possible emissions from a line that was not controlled by PRD-EL1. As mentioned earlier, results from Run 1 of the 4-in. PRD test have been discarded because an uncovered tank was being used on Line 1. Table 20 shows the summary results from the TSP samplers.

The results from the west sampler are higher than the east sampler, as expected, and the highest results are from the test with no PRD-EL1 fluid. The test with the 4-in. PRD fluid gave the best indoor air sampling results. All the results, except the west sampler with no PRD-EL1 fluid, met the previous OSHA PEL of 0.052 mg/m^3 . Again, the results of the test with no PRD-EL1 fluid are suspect because of the presence of residual PRD fluid on the surface. It is likely that these results would be higher if the PRD fluid were absent. The indoor air results indicate that the PRD-EL1 fluid reduced the fugitive emissions well below the previous OSHA PEL, but the level of reduction is uncertain. It should be pointed out that other factors also influence the indoor air chromium concentration, such as the effectiveness of the ventilation system and the air flow patterns in the building. Two of the east sampler results with PRD, but none of the west sampler results met the new finalized OSHA PEL (0.005 mg/m^3). This suggests that it is questionable whether the PRD fluid will reduce the fugitive emissions enough to meet the new PEL.

Table 20. Total Suspended Particle – Indoor Air Samples.

Test Series	4-in. PRD-EF1	2-in. PRD-EL1	No PRD-EL1
TSP Sampler Data			
TSP-West (Cr mg/m ³)	0.007	0.016	0.100
TSP-East (Cr mg/m ³)	0.004	0.005	0.019

8 Discussion, Conclusions, and Recommendations

Plating Solution and PRD Fluid Analysis

Both Phase I and Phase II results from the chemical analysis indicate that all of the plating solution samples were consistent with the baseline samples and met ANAD specifications for chromic acid, sulfuric acid, iron, and trivalent chromium. The only sample that did not meet the specifications was a baseline sample in Phase II. These results suggest that the plating solution was not adversely affected by the presence of the PRD fluid.

Although the samples of plating solution taken during Phase I appeared to be consistent, the large amount of Cr (III) in sample C1 (sample of actual PRD fluid taken because of reddish appearance) suggests an unusual reaction during plating with PRD fluid. The high levels of chromic acid in C1 suggest, however, that the sample was actually plating solution. More PRD fluid samples were taken during Phase II. The majority of Phase II samples showed no unusual amounts of chromium with the exception of sample Xa. A brown-reddish gelatinous substance was found at the surface of the tank when this sample was taken. These results suggest that occasionally the PRD fluid either becomes difficult to distinguish from the chromic acid solution or an unknown reaction between the PRD fluid and the plating solution occurs, resulting in an emulsion that changes the appearance and chemical makeup of the fluid.

Plating Quality

During Phase I, the majority of the PRD-processed components passed all qualitative (adhesion, quality, porosity) requirements. However, both PRD-processed coupons failed qualitative requirements, while the conventionally plated coupon passed these requirements. This 100 percent failure rate (2/2) suggests that the coupons may have responded differently to the plating processes than the actual components (2/8, or 25 percent failure rate of PRD-processed components). During Phase II, the

majority of the PRD-processed components did not pass all qualitative (adhesion, quality, porosity) requirements. All but three of the components/coupons failed the adhesion testing. This could have been due to insufficient cleaning of the pieces before plating.

The Phase I quantitative results (thickness, microhardness) suggest that the plating of the CCAD parts and the characteristics of the resulting electrodeposited chromium were influenced by the use of the PRD process. Quantitative results on the components produced by ANAD did not show any consistent trends when comparing PRD versus conventional processing. Phase II quantitative results on the components produced by ANAD also did not show any consistent trends, with the exception of thickness, when comparing PRD versus conventional processing. The thickness was similar on each component, but not uniform within the plating conditions. The baseline thickness was consistently equal to or greater than that of its PRD-processed counterparts.

If test coupons are being considered for qualifying plated components (by lot, etc.), then the cause of variation in response between coupons and components to the PRD process must be determined. It did not appear that the coupons reacted similarly to the components when both were subjected to the same PRD process. Additional components and coupons should be plated and evaluated using both conventional and PRD processes.

Additional testing, such as hydrogen embrittlement testing (in accordance with ASTM F 519), should be performed to further characterize the chromium plating deposited by the various processes. Hydrogen testing was not performed for this analysis because the samples that were supplied were not manufactured according to the requirements of the standard. Also, considering that stainless steel failed the adhesion testing, additional testing should be performed on substrates other than alloy steel.

The small quantity of components/coupons plated and used for this evaluation limits the accuracy and value of the results. If the Zero Emissions System is being considered for full-time implementation, additional testing is recommended. This testing should involve larger lots of parts plated under numerous plating cycles using the PRD process.

Air Quality

Air quality test results indicate that the PRD-EL1 fluid will reduce chromium emissions when placed over an existing chromium electroplating bath. The stack results indicate that, when the PRD-EL1 was in place, the chromium emissions were reduced to a level below the NESHAP standard. The baseline result without PRD-EL1 also met the standard, but those results are questionable due to residual PRD-EL1. The level of reduction is therefore unknown.

Indoor air sampling results indicate that PRD-EL1 reduced the chromium concentration in the indoor air to well below the previous OSHA PEL, but the level of reduction is again uncertain because of the suspect baseline test. Further testing is needed to confirm these results and to quantify the level of emission reduction caused by the PRD fluid. Since these tests, OSHA has finalized a new PEL for hexavalent chromium of 0.005 mg/m^3 on an 8-hour time-weighted average (the current PEL is 0.052 mg/m^3 [69FR 59305]). The results from this test indicate that it is questionable whether the new PEL would be met using the Zero Emissions System.

Operational Issues

Maintenance probably will not be limited to monitoring/adjusting the fluid layer. When parts and anodes are removed from the tank, some PRD fluid is retained (drag-out) and falls to the anode and cathode bars. These bus bars would have to be cleaned between loads to ensure proper contact with the parts hangers, because the PRD fluid is essentially a nonconductor. Improper contact could substantially alter the current plating density between the anodes and the parts being plated, thus affecting plating performance and quality. For large chromium plating baths, this cleaning operation may be difficult to perform.

Initially it was suggested that the PRD fluid drag-out could be easily removed from parts via simple washing with water. Observations made during the technology demonstration revealed that some residual PRD fluid remained on parts after washing. This residue was not quantified, but could be removed during a step after the plating process in which the parts are lowered into a solvent bath. The effect that this residue has on the solvent bath either needs to be determined or a procedure put in place that would eliminate drag-out. To make a reasonable cost analysis, long-term tracking and testing would have to be performed to determine the effects of drag-out and any associated disposal costs.

The design of this system is simple and the materials used for implementation, such as valves, controls, and piping, are readily available off the shelf, having been proven in the commercial sector. The liquid pump is the only mechanical part of the system that could likely experience failure. The system uses a proprietary material manufactured by PRD Tech, Inc. Additional PRD fluid can be acquired only through this company. This single-source restriction poses a risk if PRD Tech, Inc. should no longer produce the fluid.

References

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- ASTM B 578-87, "Standard Test Method for Microhardness of Electroplated Coatings."
- ASTM F 519-97e2, "Standard Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating Processes and Service Environments."
- Federal Register, Vol. 60, p 4948, "National Emission Standards for Chromium Emissions from Hard and Decorative Chromium Electroplating and Chromium Anodizing Tanks."
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- Society of Automotive Engineers (SAE), AMS-QQ-C-320, Chromium Plating (Electrodeposited), 1 July 2000, SAE International, www.sae.org.
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- U.S. Code of Federal Regulations, Title 29, Part 1910, Section 94, "Occupational Exposure to Hexavalent Chromium." Department of Labor, Occupational Safety and Health Administration (OSHA).
- _____, Parts 1910, 1915, et al. "Occupational Exposure to Hexavalent Chromium; Final Rule." Department of Labor, OSHA: February 28, 2006, p 10100.

Appendix A: Chromium Sample Results (Phase I)

Tables A-1 through A-4 show the chromium sample results from Phase I.

**Table A-1. Chromium sample results (CrO_3 (g/l))
Phase I.**

Sample	CrO_3 (g/l)		
	(low)	(average)	(high)
Baseline	231.31	231.53	231.75
A	235.88	236.10	236.32
B	236.27	236.48	236.69
C	235.97	236.35	236.73
C1	229.88	230.26	230.64
D	232.09	232.30	232.51
E	231.68	231.91	232.14
F	232.46	232.68	232.90

**Table A-2. Chromium sample results H_2SO_4 (g/l)
Phase I.**

Sample	H_2SO_4 (g/l)		
	(low)	(average)	(high)
Baseline	2.58	2.60	2.62
A	2.62	2.64	2.66
B	2.67	2.68	2.69
C	2.67	2.68	2.69
C1	2.51	2.53	2.55
D	2.54	2.58	2.62
E	2.60	2.62	2.64
F	2.62	2.63	2.65

Table A-3. Chromium sample results [Fe(⁹/I)] Phase I.

Sample	Fe(⁹/I) (average)
Baseline	2.891
A	2.853
B	2.809
C	2.797
C1	2.818
D	2.707
E	2.697
F	2.682

Table A-4. Chromium sample results [CR(III)] Phase I.

Sample	CR(III) (mg/ml)		
	(low)	(average)	(high)
Baseline	1.498	1.502	1.506
A	1.636	1.640	1.644
B	1.503	1.507	1.511
C	1.491	1.499	1.507
D	1.516	1.525	1.534
E	1.245	1.247	1.249
F	1.558	1.578	1.598
C1	5.417	5.427	5.437

Appendix B: Chromium Sample Results (Phase II)

Tables B-1 through B-4 show the chromium sample results from Phase II.

**Table B-1. Chromium sample results CrO_3 (g/l)
Phase II.**

Sample	CrO_3 (g/l)		
	(low)	(average)	(high)
A	204.48	204.5	204.60
C	205.65	205.7	205.71
E	205.52	205.6	205.58
G	205.04	205.0	205.04
I	205.76	205.8	205.86
K	205.60	205.7	205.76
M	152.55	152.6	152.61
N	193.07	193.1	193.13
O	190.66	190.7	190.72

**Table B-2. Chromium sample results H_2SO_4 (g/l)
Phase II.**

Sample	H_2SO_4 (g/l)		
	(low)	(average)	(high)
A	2.28	2.32	2.36
C	2.26	2.29	2.32
E	2.26	2.32	2.38
G	2.13	2.22	2.31
I	2.06	2.22	2.38
K	2.22	2.25	2.28
M	1.66	1.70	1.74
N	2.06	2.11	2.16
O	2.12	2.13	2.14

**Table B-3. Chromium sample results Fe(g/l)
Phase II.**

Sample	Fe(g/l)		
	(low)	(average)	(high)
Xa		112	
B		0.0002	
D		0.4	
F		0.04	
H		0.002	
J		54.4	
L		0.4	

**Table B-4. Chromium sample results Cr(g/l)
Phase II.**

Sample	Cr(g/l)		
	(low)	(average)	(high)
Xa		10.56	
B		0.018	
D		0.740	
F		0.010	
H		0.02	
J		3.684	
L		1.088	

Appendix C: Materials Laboratory Report (Phase I)

AMSTA-AR-CCB-EA

4 December 2002

MEMORANDUM FOR AMSTA-AR-CCB-EB

1. Background

Materials Engineering was requested by John Cannon, Process Engineering Team, to conduct testing on thirteen components and three test coupons (16 pieces, total) supplied to Benét Laboratories by Anniston Army Depot (ANAD) and Corpus Christi Army Depot (CCAD). Eight components and two coupons had been Chromium Electroplated at these installations using PRD Tech Inc.'s *Zero Emission Process*. This process involves using an immiscible liquid layer across the top of the chromic acid electroplating bath to eliminate aerosol emissions from the tank during plating. Two different layer thicknesses were used in plating; 2" and 4". The remaining components and coupons were conventionally electroplated in accordance with each installation's normal procedures. Our tests were performed in order to characterize the resulting chromium electroplating for all components/coupons, and then compare the results to determine what effects the Zero Emission Process (commonly referred to as the PRD-process) had on the electroplating. Our testing was conducted in accordance with AMS 2406K ("Plating, Chromium Hard Deposit"), para. 4.2.3) and AMS-QQ-C320 ("Chromium Plating"), para. 3.4.2 and 4.5.4). The supplied components and coupons were identified as follows (Table C-1):

Table C-1. Components and coupons.

COMPONENTS AND COUPONS			
114L2425-1 Baseline	12304148 Baseline	12286191 2" PRD	12284387 4" PRD
114L2425-1 2" PRD	12304148 2" PRD	12286191 4" PRD	Baseline coupons
145H7359-1 Baseline	12304148 4" PRD	12284387 Baseline	2" PRD coupons
145H7359-1 4" PRD	12286191 Baseline	12284387 2" PRD	4" PRD coupons

Components 114L2425-1 and 145H7359 -1 were produced at CCAD, while all the remaining components and coupons were produced at ANAD. The components/coupons were subjected to three different chromium plating conditions or lots: baseline (conventional) plating, 2" PRD fluid plating, and 4" PRD fluid plating. All components were plated for twelve hours duration at ANAD's minor plating facility.

2. Procedure

Each component and coupon was subjected to the following tests/evaluations:

- a. Thickness Testing in accordance with ASTM B487
- b. Adhesion Testing in accordance with ASTM B571 (para. 8)
- c. Quality Testing in accordance with AMS 2406K (para. 3.5)
- d. Porosity Testing in accordance with AMS-QQ-C-320
- e. Hardness Testing in accordance with ASTM B578

3. Results/Discussion

a. Thickness Testing

Chromium plating thickness was measured by examining a metallographic cross-section of the component/coupon in accordance with ASTM B487. Multiple cross-sections were obtained from each component/coupon. These cross-sections were mounted, polished, and etched with a 2 percent nital solution. Please note that Component #145H7359-1 did not etch with nital. An X-Ray fluorescent chemical analysis was performed on the base metal of this piece, and it was qualitatively identified as a stainless steel. All other components were successfully etched, indicating the base material was a steel alloy.

The thickness of the chromium plating for each test piece is shown in Table C-2. Measurements were taken at multiple points on each sample and averaged. We are unaware of any specific requirement(s) for plating thickness on the components/coupons, so the values are reported mainly for comparing the plating processes.

Table C-2. Results of thickness testing.

Part Identification	Plating Thickness (inches)
114L2425-1 Baseline	0.0113
114L2425-1 2 inch	0.0040
145H7359-1-Baseline	0.0137
145H7359-1- 4 inch	0.0126
12304148 Baseline	0.0074
12304148 2 inch	0.0070
12304148 4 inch	0.0094
12286191 Baseline	0.0232
12286191 2 inch	0.0131
12286191 4 inch	0.0129
12284387 Baseline	0.0149
12284387 2 inch	0.0097
12284387 4 inch	0.0183
Baseline Coupon	0.0038
2" PRD Coupon	0.0031
4" PRD Coupon	0.0060

Figure C-1 is an example of a cross-section used for thickness measurements.

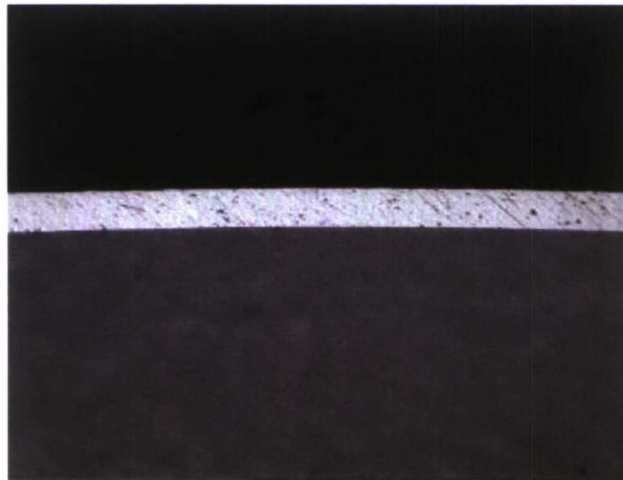


Figure C-1. Typical view of chromium plating sample used for thickness measurement.

b. Adhesion Testing

Adhesion testing was conducted in accordance with ASTM B571 (para. 8). The Grind Saw Test was performed on eight areas for each specimen, and the areas were then examined at 5X magnification. Satisfactory adhesion was observed in all of the specimens except 145H7359-1 Baseline, 2" PRD Coupon, and 4" PRD Coupon. Results from this test can be viewed in Table C-3.

Table C-3. Results of adhesion testing.

Part Identification	Adhesion (Pass/Fail)
114L2425-1 Baseline	Pass
114L2425-1 2" PRD	Pass
145H7359-1 Baseline	<i>Fail</i>
145H7359-1 4" PRD	Pass
12304148 Baseline	Pass
12304148 2" PRD	Pass
12304148 4" PRD	Pass
12286191 Baseline	Pass
12286191 2" PRD	Pass
12286191 4" PRD	Pass
12284387 Baseline	Pass
12284387 2" PRD	Pass
12284387 4" PRD	Pass
Baseline Coupons	Pass
2" PRD Coupons	<i>Fail</i>
4" PRD Coupons	<i>Fail</i>

Representative photographs of a good adhesion sample (Figure C-1) and the failed samples are displayed in Figures C-2 through C-5.

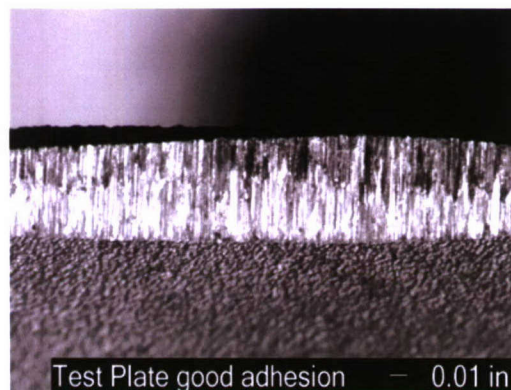


Figure C-2. An example of a sample that passed adhesion criteria.

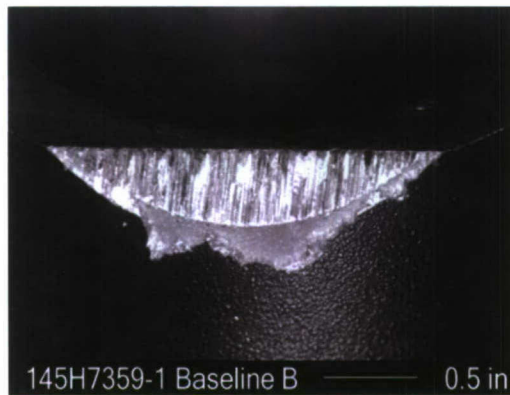


Figure C-3. Sample 145H7359-1 Baseline that failed the adhesion test due to the lifting/peeling of the chromium plating.

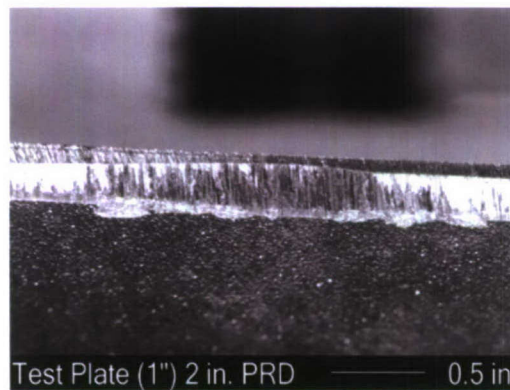


Figure C-4. 2" PRD Coupon that failed the adhesion test.

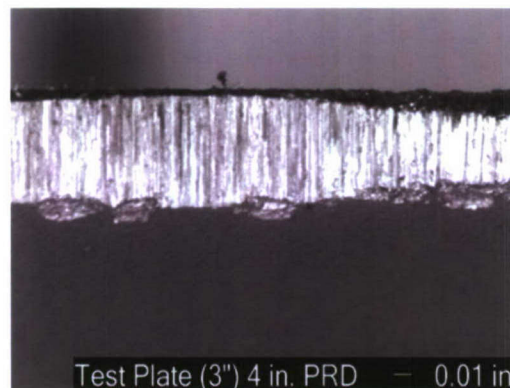


Figure C-5. 4" PRD Coupon that failed the adhesion test.

E. Quality Testing

Quality testing was performed on each piece (components, coupons) by visual inspection in accordance with AMS 2406K (para. 3.5). The specification requires that the plating be firmly bonded to the base metal, while maintaining smooth and uni-

form appearance. Pinholes and other small imperfections that resulted from the plating not filling in defects on the surface metal are considered acceptable. A magnification of 5X was used to inspect the pieces. The results can be seen in Table C-4.

Table C-4. Results of quality testing.

Part Identification	Quality (Pass/Fail)
114L2425-1 Baseline	Pass
114L2425-1 2" PRD	Pass
145H7359-1 Baseline	Pass
145H7359-1 4" PRD	Pass
12304148 Baseline	Pass
12304148 2" PRD	Fail
12304148 4" PRD	Pass
12286191 Baseline	Pass
12286191 2" PRD	Pass
12286191 4" PRD	Pass
12284387 Baseline	Pass
12284387 2" PRD	Pass
12284387 4" PRD	Fail
Baseline Coupons	Pass
2" PRD Coupons	Pass
4" PRD Coupons	Pass

As seen in this table, two pieces, 12304148 2" PRD and 12284387 4" PRD, failed this test. Both exhibited a burst "bubble" in the plating surface (see Figures C-6 & C-7).

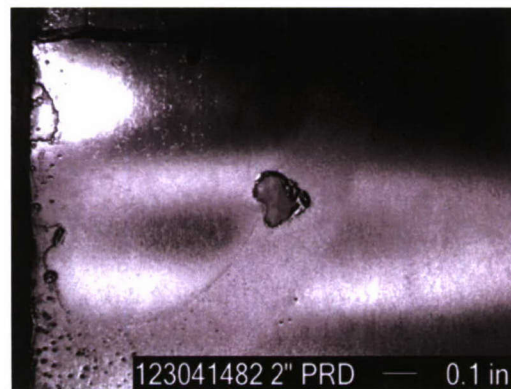


Figure C-6. The ruptured "bubble" in sample 12304148 2" PRD.

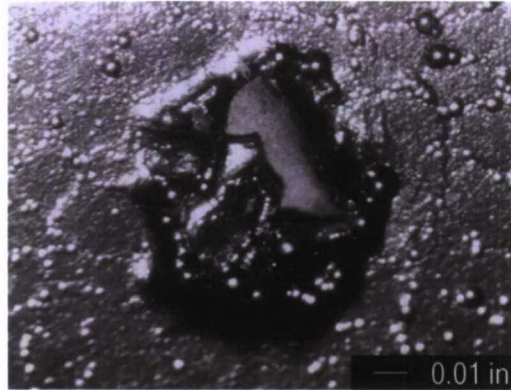


Figure C-7. The ruptured "bubble" in sample 12284387 4" PRD.

d. Porosity Testing

Porosity testing was conducted in accordance with AMS-QQ-C-320. A ferroxyl test was completed by soaking filter paper in ferroxyl solution and heating to 180-200 degrees F. The paper was then placed over the entire plated area on each piece for ten minutes. Blue markings occurred on the filter paper as a result of the reaction of the base metal with the solution. These markings are indicative of porosity. The results of this test can be seen in Table C-5.

Table C-5. Results of porosity testing.

Part Identification	Porosity (Pass/Fail)
114L2425-1 Baseline	Pass
114L2425-1 2" PRD	Pass
145H7359-1 Baseline	Pass
145H7359-1 4" PRD	Pass
12304148 Baseline	Pass
12304148 2" PRD	Pass
12304148 4" PRD	Pass
12286191 Baseline	Pass
12286191 2" PRD	Pass
12286191 4" PRD	Pass
12284387 Baseline	Pass
12284387 2" PRD	Pass
12284387 4" PRD	Pass
Baseline Coupons	Pass
2" PRD Coupons	Pass
4" PRD Coupons	Pass

E. Hardness Testing

Hardness testing was completed in accordance with ASTM B578. A Vicker's indenter with a 100 gram load was used. The minimum acceptable hardness as per the standard was 700 HV. The average hardness for all samples was above the 700 HV minimum (Table C-6). Additionally, there were no individual hardness readings below 700 HV.

Table C-6. Results from Vickers Microhardness Testing.

Part Identification	Avg. Microhardness Value (HV)
144L2425-1 Baseline	906.90
114L2425-1 2"	824.20
145H7359-1 Baseline	921.33
145H7359-1 4"	885.87
1230418- Baseline	855.33
12304148- 2"	858.60
12304148- 4"	858.60
12286191- Baseline	877.07
12286191- 2"	885.47
12286191- 4"	894.40
12284387- Baseline	885.33
12284387- 2"	856.07
12284387- 4"	835.07
Baseline Coupon	844.87
2" PRD Coupon	837.40
4" PRD Coupon	893.00

4. Summary

The results from our testing are summarized and displayed in Table C-7.

Table C-7. Summary of results.

Part Identification	Thickness (Inches)	Adhesion (Pass/Fail)	Quality (Pass/Fail)	Porosity (Pass/Fail)	Microhardness (HV)
114L2425-1 Baseline	0.0113	Pass	Pass	Pass	Pass (906.90)
114L2425-1 2" PRD	0.0040	Pass	Pass	Pass	Pass (824.20)
145H7359-1 Baseline	0.0137	Fail	Pass	Pass	Pass (921.33)
145H7359-1 4" PRD	0.0126	Pass	Pass	Pass	Pass (885.87)
12304148 Baseline	0.0074	Pass	Pass	Pass	Pass (855.33)
12304148 2" PRD	0.0070	Pass	Fail	Pass	Pass (858.60)
12304148 4" PRD	0.0094	Pass	Pass	Pass	Pass (858.60)
12286191 Baseline	0.0232	Pass	Pass	Pass	Pass (877.07)
12286191 2" PRD	0.0131	Pass	Pass	Pass	Pass (885.47)
12286191 4" PRD	0.0129	Pass	Pass	Pass	Pass (894.40)
12284387 Baseline	0.0149	Pass	Pass	Pass	Pass (885.33)
12284387 2" PRD	0.0097	Pass	Pass	Pass	Pass (856.07)
12284387 4" PRD	0.0183	Pass	Fail	Pass	Pass (835.07)
Baseline Coupons	0.0038	Pass	Pass	Pass	Pass (844.87)
2" PRD Coupons	0.0031	Fail	Pass	Pass	Pass (837.40)
4" PRD Coupons	0.0060	Fail	Pass	Pass	Pass (893.00)

Table C-7 shows that three of the thirteen components, and two of the three coupons failed at least one test. One of the component failures was actually a component subjected to the conventional electroplating process. Additionally, there was a noticeable decrease in the microhardness and thickness of the plating in components PRD-processed (114L2425-1 and 145H77359-1 series) when compared to their conventionally plated counterpart components. This decrease was more pronounced in component 114L2425-1 2" PRD.

5. Conclusions

- The majority of the PRD-processed components passed all qualitative (adhesion, quality, porosity) requirements.
- Both PRD-processed coupons failed qualitative requirements; the conventionally plated coupon passed these requirements. This 100% failure rate (2/2) suggests that the coupons may have responded differently to the plating processes than the actual components (2/8, or 25% failure rate of PRD-processed components).
- The quantitative results (thickness, microhardness) suggest that the plating of the CCAD parts and the characteristics of the resulting electrodeposited Chromium, were influenced by the use of the PRD-process.

- Quantitative results on the components produced by ANAD did not show any consistent trends when comparing PRD versus conventional processing.

6. Recommendations

Additional testing, such as hydrogen testing (in accordance with ASTM F 519), should be performed to further characterize the Chromium plating deposited by the various processes. Hydrogen testing was not performed for this analysis because the samples that were supplied were not manufactured according to the requirements of the standard. Also, considering the stainless steel failed the adhesion testing, additional testing should be performed on substrates other than alloy steel.

If test coupons are being considered for qualifying plated components (by lot, etc.), then the cause of variation in response between coupons and components to the PRD-process must be determined. Currently, it does not appear that the coupons are reacting similarly to the components when both are subjected to the same PRD-process.

Additional components and coupons should be plated and evaluated using both conventional and PRD processes. The low quantity of components/coupons plated and used for this evaluation limits the accuracy of our results.

Ashley Fiegel

Materials Engineering Team

Appendix D: Materials Laboratory Report (Phase II)

AMSTA-AR-CCB-EA

6 August 2003

MEMORANDUM FOR AMSTA-AR-CCB-EB

1. Background

Materials Engineering Team was requested by John Cannon, Process Engineering Team, to conduct testing on thirteen components and three test coupons (15 pieces, total) supplied to Benét Laboratories by Anniston Army Depot (ANAD). Four components and six coupons had been Chromium Electroplated at this installation using PRD Tech Inc.'s *Zero Emission Process*. This process involves using an immiscible liquid layer across the top of the chromic acid electroplating bath to eliminate aerosol emissions from the tank during plating. Two different layer thicknesses were used during plating; 2" and 4". The remaining components and coupons were conventionally electroplated in accordance with the installation's normal procedures. Our tests were performed in order to characterize the resulting chromium electroplating for all components/coupons, and then compare the results to determine what effects the Zero Emission Process (commonly referred to as the PRD-process) had on the electroplating. Our testing was conducted in accordance with AMS 2406K ("Plating, Chromium Hard Deposit"), para. 4.2.3) and AMS-QQ-C320 ("Chromium Plating"), para. 3.4.2 and 4.5.4). The supplied components and coupons were identified as follows (Table D-1):

Table D-1. Components and coupons.

COMPONENTS AND COUPONS		
12304148 Baseline	12286191 Baseline	Baseline coupons
12304148 2" PRD	12286191 2" PRD	2" PRD coupons
12304148 4" PRD	12286191 4" PRD	4" PRD coupons

All components and coupons were produced at ANAD. The components/coupons were subjected to three different chromium plating conditions or lots: baseline (conventional) plating, 2" PRD fluid plating, and 4" PRD fluid plating. All components were plated for twelve hours duration at ANAD's minor components plating facility.

2. Procedure

Each component and coupon was subjected to the following tests/evaluations:

- a. Thickness Testing in accordance with ASTM B487
- b. Adhesion Testing in accordance with ASTM B571 (para. 8)
- c. Quality Testing in accordance with AMS 2406K (para. 3.5)
- d. Porosity Testing in accordance with AMS-QQ-C-320
- e. Hardness Testing in accordance with ASTM B578

3. Results/Discussion

a. Thickness Testing

Chromium plating thickness was measured by examining a metallographic cross-section of the component/coupon in accordance with ASTM B487. Multiple cross-sections were obtained from each component/coupon. These cross-sections were mounted, polished, and etched with a 2% nital solution.

The thickness of the chromium plating for each test piece is shown in Table D-1. Measurements were taken at multiple points on each sample and averaged. We are unaware of any specific requirement(s) for plating thickness on the components/coupons, so the values are reported mainly for comparing the varied plating processes.

Table D-2. Results of thickness testing.

Part Identification	Plating Thickness (inches)
12304148 Baseline	0.006
12304148 2"	0.003
12304148 4"	0.006
12286191 Baseline	0.017
12286191 2"	0.013
12286191 4"	0.013
Baseline Coupon	0.002 see note

2" PRD Coupon	0.001 see note
4" PRD Coupon	0.001 see note

Note: The average Thickness for 2" and 4" PRD was below the minimum RECOMMENDED thickness requirement of AMS 2406K (0.002"). Additionally all three coupon sets had a rough substrate surface finish compared to the actual components. This rougher surface appeared to deleteriously affect the plating process and resulting plating.

An example of a cross-section used for thickness measurements can be seen in Figure D-1.

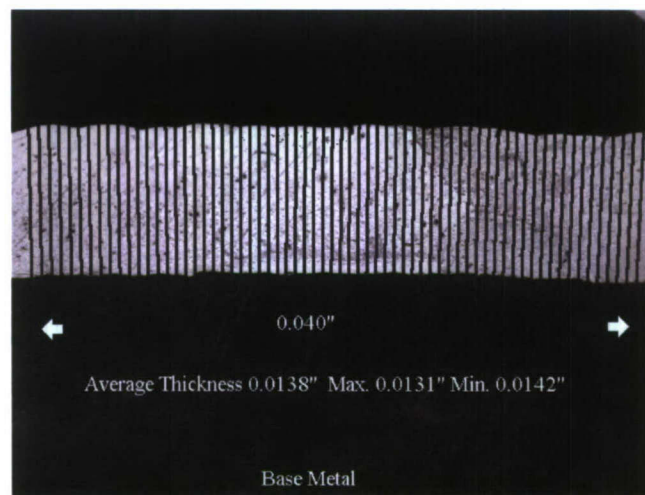


Figure D-1. Typical view of chromium plating sample used for thickness measurement.

An example of a cross-section showing thickness and substrate finish can be seen in Figure D-2.

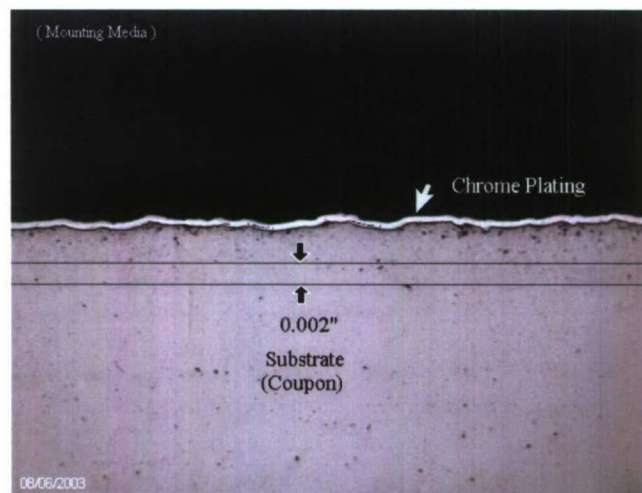


Figure D-2. Baseline Coupon with thin plating and rough substrate surface finish.

b. Adhesion Testing

Adhesion testing was conducted in accordance with ASTM B571 (para. 8). The Grind Saw Test was performed on eight areas for each specimen, and the areas were then examined at 5X magnification.

Results from this test can be viewed in Table D-2.

Table D-3. Results of adhesion testing.

Part Identification	Adhesion (Pass/Fail)
12304148 Baseline	Fail
12304148 2" PRD	Fail
12304148 4" PRD	Fail
12286191 Baseline	Pass
12286191 2" PRD	Pass
12286191 4" PRD	Pass
Baseline Coupons	Fail
2" PRD Coupons	<i>Fail</i>
4" PRD Coupons	<i>Fail</i>

The specification states that lifting or peeling of the coating is evidence of unsatisfactory adhesion.

Representative photographs of failed samples are displayed in Figures D-3 to D-6.

Figures D-3 to D-6 showing failed samples.

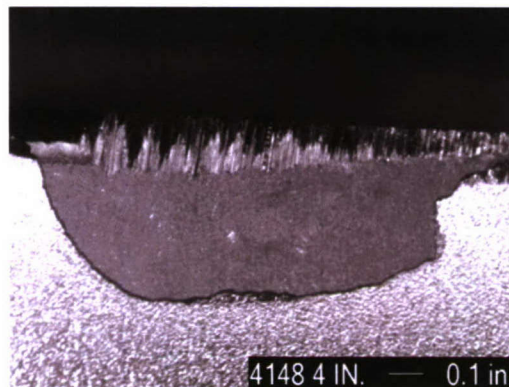


Figure D-3. Sample (4148 4 in).



Figure D-4. Sample (4148 Baseline).

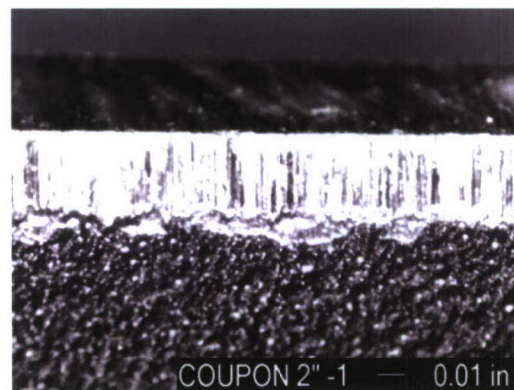


Figure D-5. Sample (Coupon 2'').



Figure D-6. Sample (Coupon Baseline).

c. Quality Testing

Quality testing was performed on each piece (components, coupons) by visual inspection in accordance with AMS 2406K (para. 3.5). The specification requires that the plating be firmly bonded to the base metal, while maintaining smooth and uniform appearance. Pinholes and other small imperfections that resulted from the plating not filling in defects on the surface metal are considered acceptable. A mag-

nification of 5X was used to inspect the pieces. The results can be seen in Table D-3.

Table D-4. Results of quality testing.

Part Identification	Quality (Pass/Fail)
12304148 Baseline	Pass
12304148 2" PRD	Pass
12304148 4" PRD	Fail
12286191 Baseline	Fail
12286191 2" PRD	Pass
12286191 4" PRD	Pass
Baseline Coupons	Pass
2" PRD Coupons	Pass
4" PRD Coupons	Pass

Coupon 12304148 4" has several small surface pits (not through to base metal) and 12286191 Baseline has visible pimples, hairline surface cracks (not through to base metal) and a generally rough texture.

d. Porosity Testing

Porosity testing was conducted in accordance with AMS-QQ-C-320. A ferroxyl test was completed by soaking filter paper in ferroxyl solution and heating to 180-200 °F. The paper was then placed over the entire plated area on each piece for ten minutes. Blue markings occurred on the filter paper as a result of the reaction of the base metal with the solution. These markings are indicative of porosity. The results of this test can be seen in Table D-4.

Table D-5. Results of porosity testing.

Part Identification	Porosity (Pass/Fail)
12304148 Baseline	Pass
12304148 2" PRD	Pass
12304148 4" PRD	Pass
12286191 Baseline	Pass
12286191 2" PRD	Pass
12286191 4" PRD	Pass
Baseline Coupons	Pass
2" PRD Coupons	Fail
4" PRD Coupons	Fail

E. Hardness Testing

Hardness testing was completed in accordance with ASTM B578. A Vicker's indenter with a 100-gram load was used. The minimum acceptable hardness as per the standard was 700 HV. The average hardness for all samples was above the 700 HV minimum (Table D-5). Additionally, there were no individual hardness readings below 700 HV.

Table D-6. Results from Vickers microhardness testing.

Part Identification	Avg. Microhardness Value (HV)
1230418- Baseline	949
12304148- 2"	933
12304148- 4"	940
12286191- Baseline	929
12286191- 2"	932
12286191- 4"	947
Baseline Coupon	876
2" PRD Coupon	see Note
4" PRD Coupon	865

Note: The Hardness of the plating on Coupon 2" PRD was too thin to measure at the required HV₁₀₀ Load.

4. Summary

The results from our testing are summarized and displayed in Table D-7:

Table D-7. Summary of results.

Part Identification	Thickness (Inches)	Adhesion (Pass/Fail)	Quality (Pass/Fail)	Porosity (Pass/Fail)	Microhardness (HV)
12304148 Baseline	0.006	Fail	Pass	Pass	Pass (949.0)
12304148 2" PRD	0.003	Fail	Pass	Pass	Pass (933.0)
12304148 4" PRD	0.006	Fail	Fail	Pass	Pass (940.0)
12286191 Baseline	0.017	Pass	Fail	Pass	Pass (929.0)
12286191 2" PRD	0.013	Pass	Pass	Pass	Pass (932.0)
12286191 4" PRD	0.013	Pass	Pass	Pass	Pass (947.0)
Baseline Coupons	0.002	Fail	Pass	Pass	Pass (876.0)
2" PRD Coupons	0.001	Fail	Pass	Fail	No result- too thin
4" PRD Coupons	0.001	Fail	Pass	Fail	Pass (865.0)

Table D-7 shows that four of the six components, and the three coupons failed at least one test. All components/coupons subjected to the conventional electroplating process failed at least one of the tests.

5. Conclusions

- The majority of the PRD-processed components did not pass all qualitative (adhesion, quality, porosity) requirements.
- All but three of the components/coupons failed the adhesion testing. This could be due to insufficient cleaning of the pieces before plating.
- Quantitative results on the components produced by ANAD did not show any consistent trends, with the exception of thickness, when comparing PRD versus conventional processing. The thickness was similar on each component, but not uniform within the plating conditions. The baseline thickness was consistently equal to or greater than that of its PRD-processed counterparts.

6. Recommendations

- Additional testing, such as hydrogen testing (in accordance with ASTM F 519), should be performed to further characterize the Chromium plating deposited by the various processes. Hydrogen testing was not performed for this analysis because the samples that were supplied were not manufactured according to the requirements of the standard.
- If test coupons are being considered for qualifying plated components (by lot, etc.), then the cause of variation in response between coupons and components to the PRD-process must be determined. Currently, it does not appear that the coupons are reacting similarly to the components when both are subjected to the same PRD-process.
- The low quantity of components/coupons plated and used for this evaluation limits the accuracy of our results. If the Zero Emissions Process is being considered for full time implementation, it is Benét Materials Engineering Team's recommendation that additional testing be done. This testing should involve larger lots of parts plated under numerous plating cycles using the PRD process.

Ashley Fiegel

Materials Engineering Team

Appendix E: Photographs of Phase II Demonstration



Figure E-1. Condition of cathode bars in tank 12-b, pre-4-in. PRD Plating Start.



Figure E-2. Overall view of tank 12-b, pre-4-in. PRD plating start.



Figure E-3. Overall view from above tank 12-a, pre-4-in. PRD plating start.

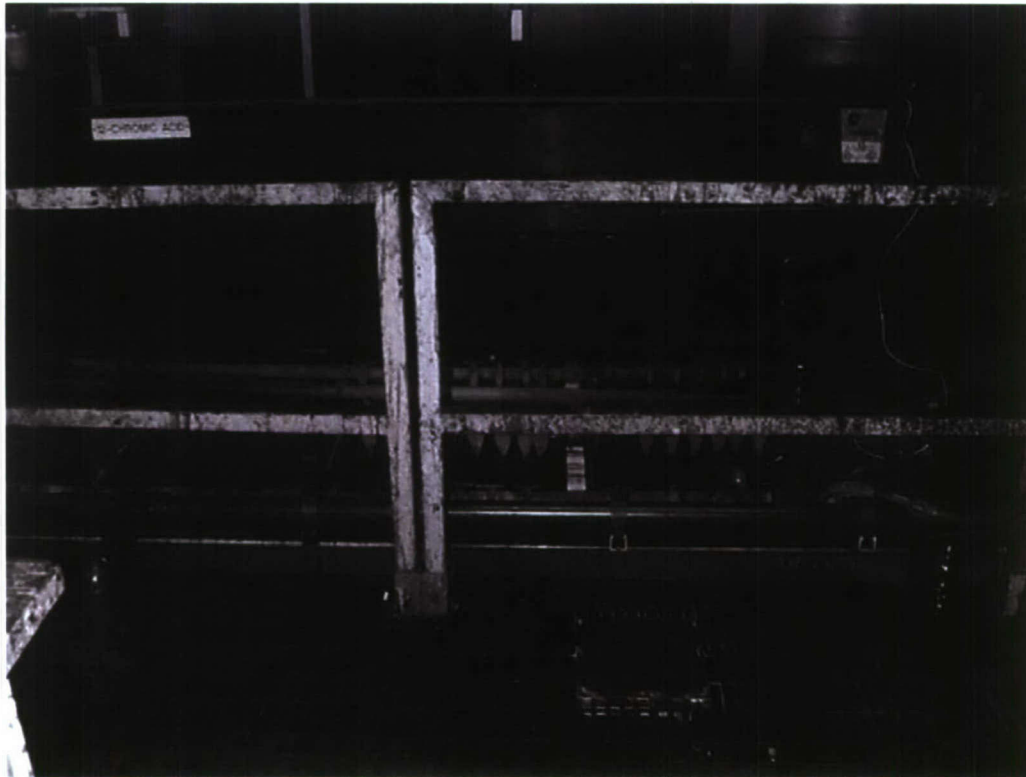


Figure E-4. Overall view from the side of tank 12-a pre-4-in. PRD plating start.



Figure E-5. Overall shot of tank 12-b, pre-4-in. at PRD plating start.



Figure E-6. Prepared samples pre-4-in. PRD plating start.



Figure E-7. Close-up of prepared IP shaft, pre-4-in. PRD plating start.



Figure E-8. Close-up of prepared adapter, pre-4-in. PRD plating start.



Figure E-9. View of tank 12-b during 4-in. PRD plating cycle.



Figure E-10. Overhead view of tank 12-a during 4-in. PRD plating cycle.



Figure E-11. Plated LP shaft at end of 4-in. PRD plating cycle.

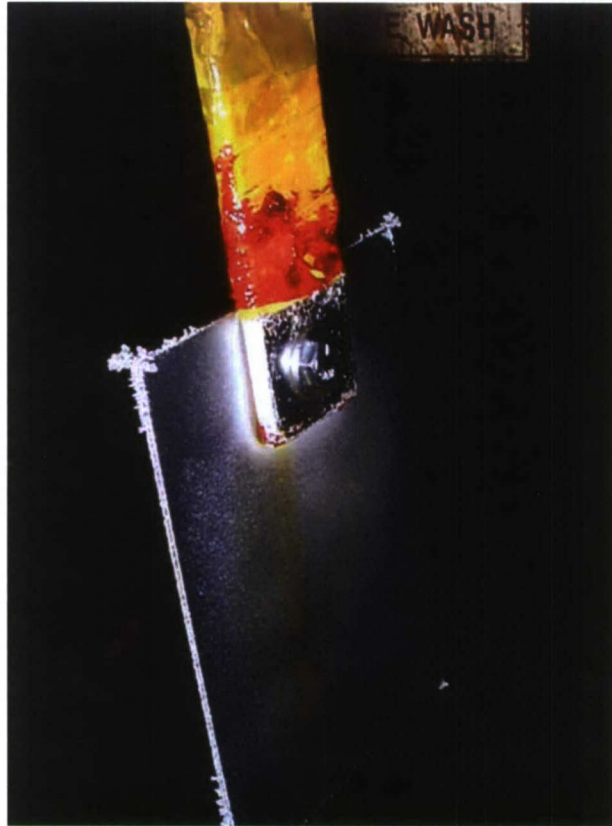


Figure E-12. Plated coupon at end of 4-in. PRD plating cycle.

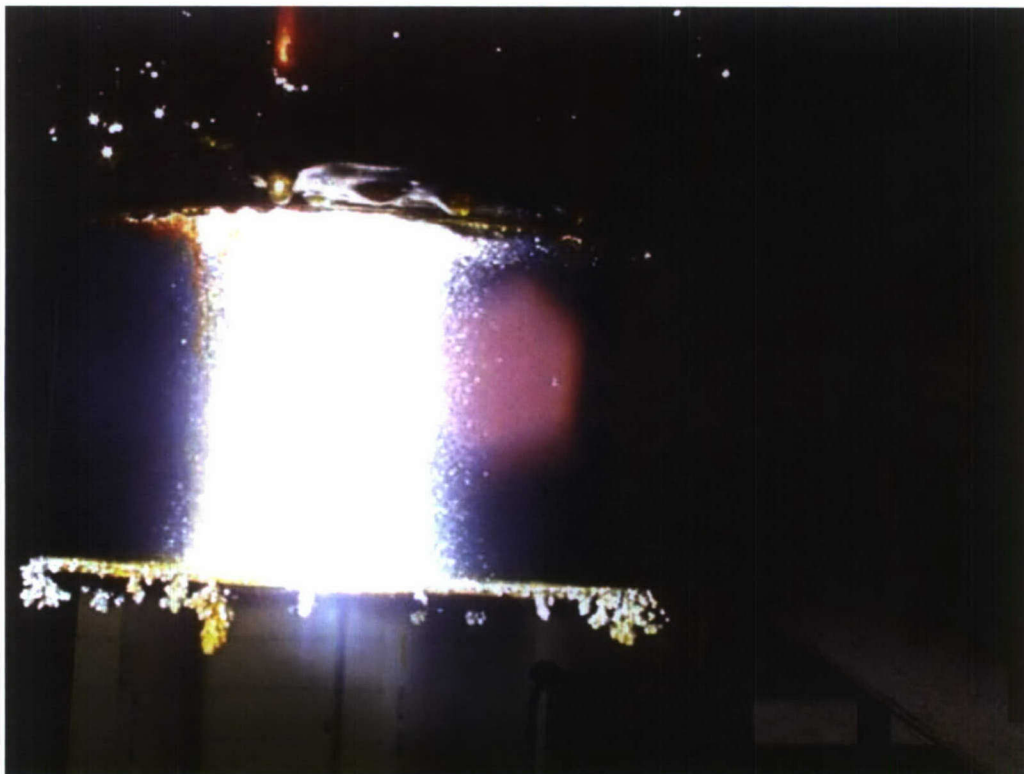


Figure E-13. Plated adaptor at end of 4-in. PRD plating cycle.



Figure E-14. Plated samples hang from rack at end of 4-in. PRD plating cycle.



Figure E-15. Prepared samples prior to 2-in. PRD plating cycle.

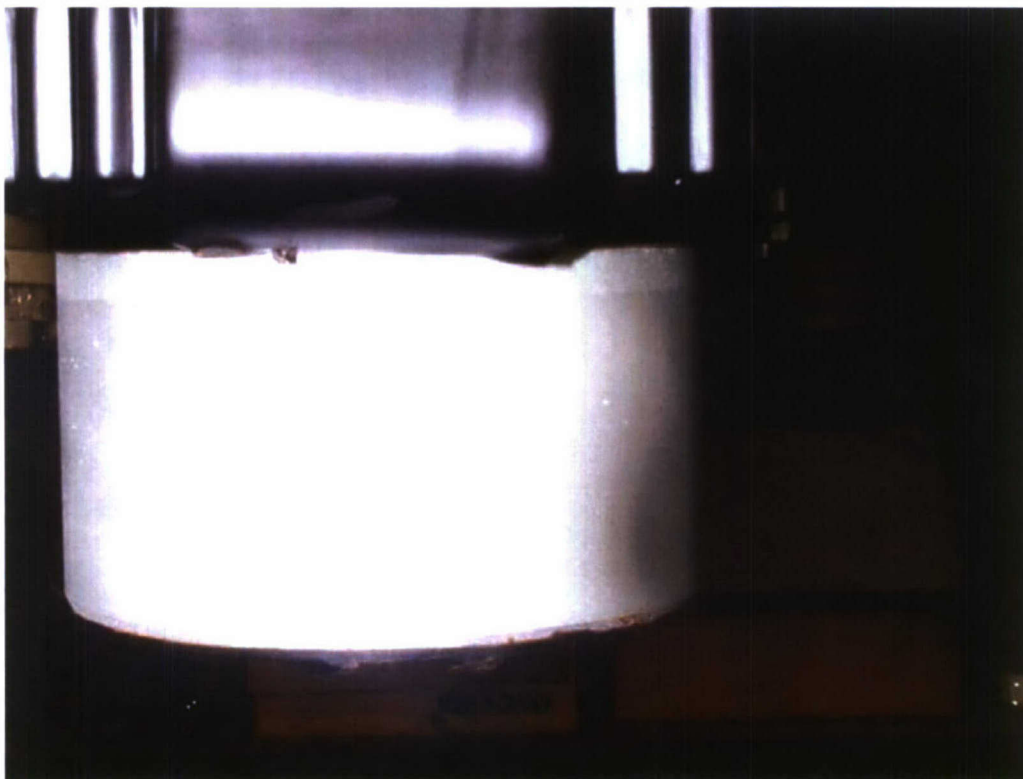


Figure E-16. Prepared adaptor prior to 2-in. PRD plating cycle.

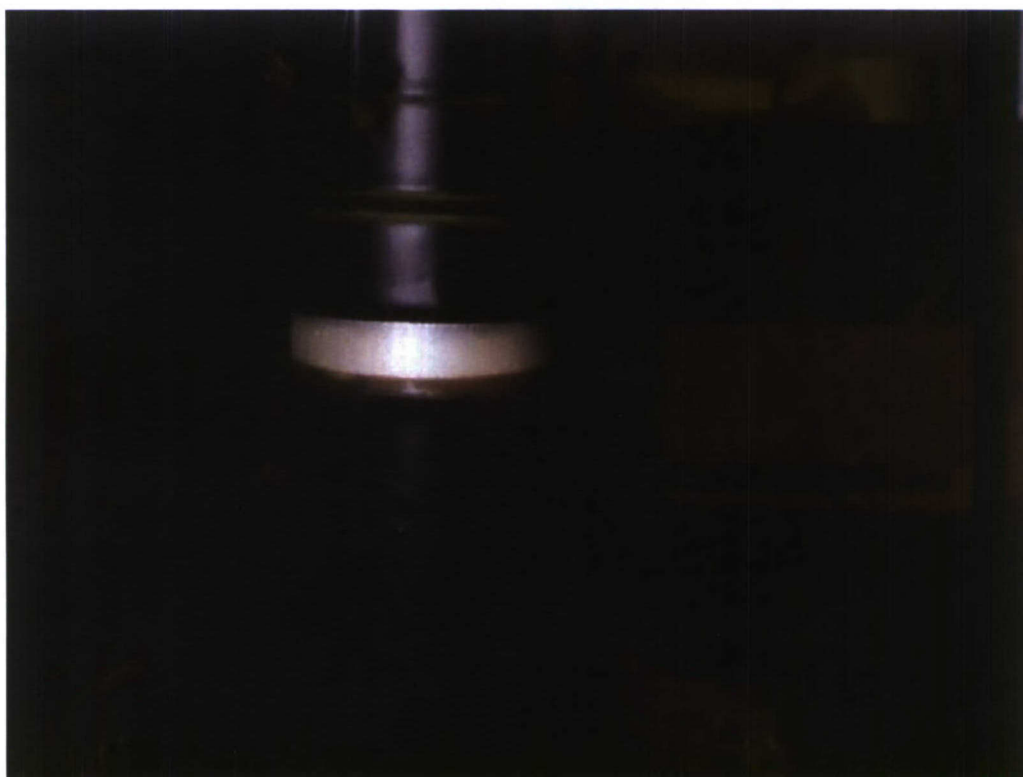


Figure E-17. Prepared IP shaft prior to 2-in. PRD plating cycle.



Figure E-18. Plated samples at end of 2-in. PRD plating cycle.



Figure E-19. Plated IP shaft at end of 2-in. PRD plating cycle.



Figure E-20. Plated coupon at end of 2-in. PRD plating cycle.

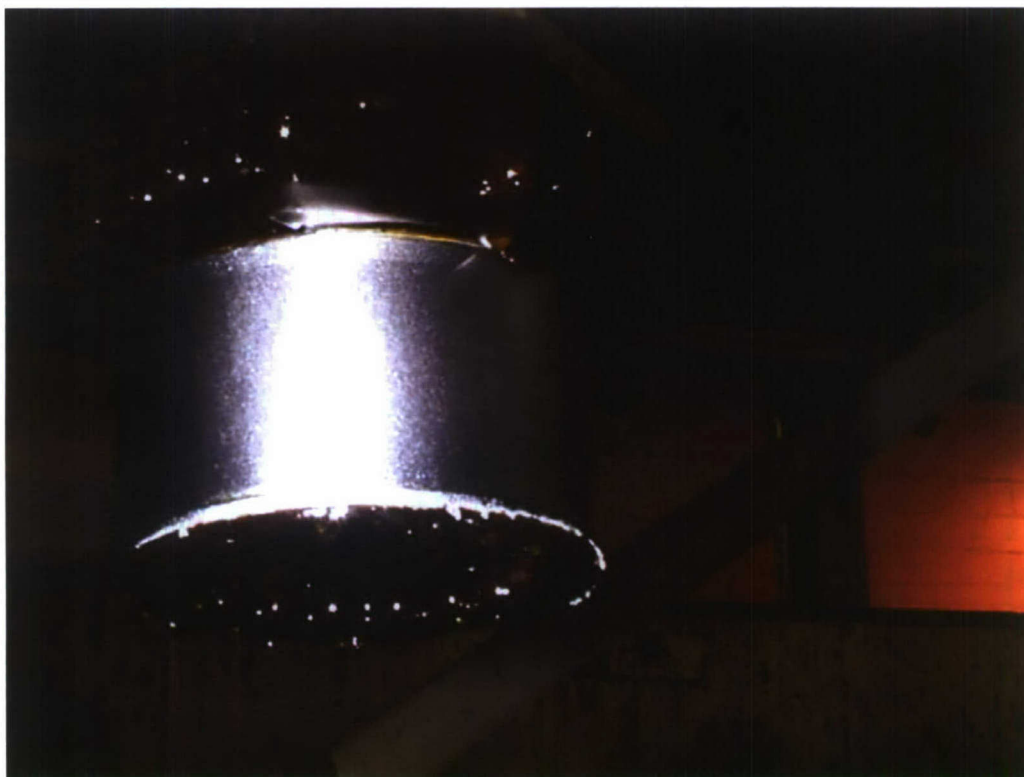


Figure E-21. Plated adaptor at end of 2-in. PRD plating cycle.



Figure E-22. Tank 12-b at end of 2-in. PRD plating cycle.



Figure E-23. Tank 12-a at end of 2-in. PRD plating cycle.



Figure E-24. Prepared parts at start of regular chromium plating cycle.



Figure E-25. Prepared adaptor, at start of regular chromium plating cycle.



Figure E-26. Prepared LP shaft at start of regular chromium plating cycle.



Figure E-27. All prepared samples at start of regular chromium plating cycle.



Figure E-28. Tank 12-b at start of regular chromium plating cycle.



Figure E-29. Tank 12-a at start of regular chromium plating cycle.

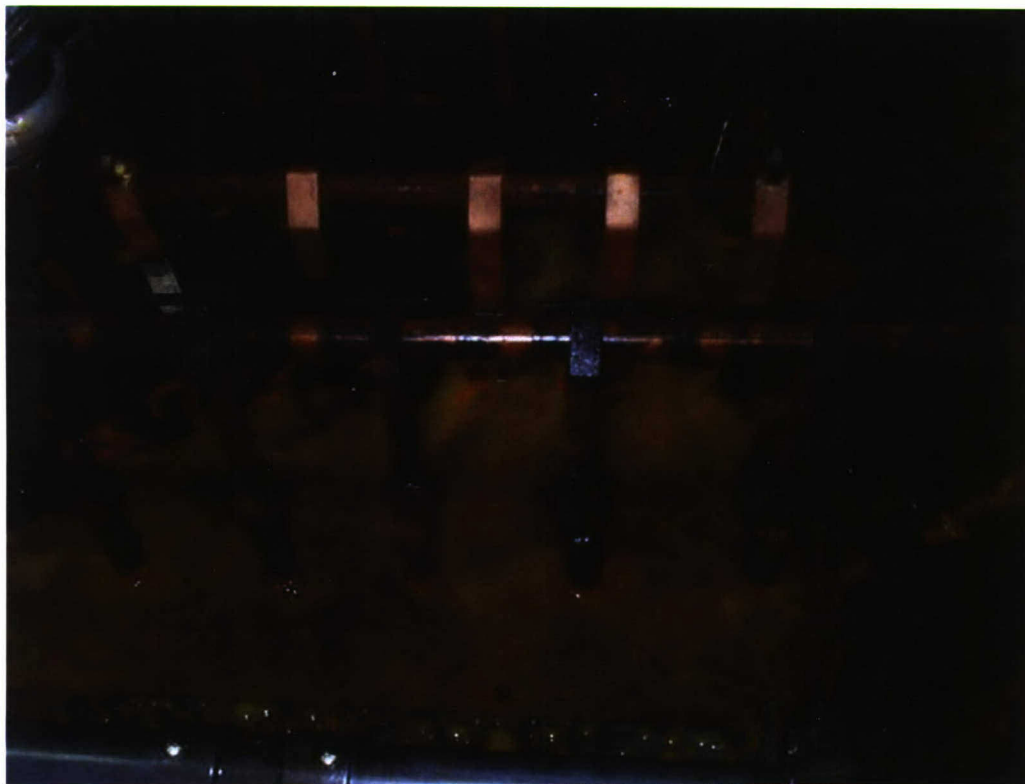


Figure E-30. Tank 12-b at end of regular chromium plating cycle.



Figure E-31. Plated IP shaft at end of regular chromium plating cycle.



Figure E-32. Plated adaptor at end of regular chromium plating cycle.



Figure E-33. Plated parts at end of regular chromium plating cycle.

Appendix F: Benét Laboratories Field Book

PRD ZERO EMISSIONS PROJECT.

Monday, August 26, 2002

0800 HRS. ARRIVE AT ANNISDON Army DEPOT (A

0930 HRS. RECEIVE BADGE FOR ADMITTANCE,
HOWEVER, MAX PHELPS (PAWL) NEEDS
TO GO TO DIFFERENT AREA TO RECEIVE
BADGE.

1000 HRS. PERIOD AT Tony POWARDS OFFICE,
JEREMY TURNER (AAD POINT OF CONTACT)
IS AT MAIN GATE ESCORTING TWO
GENTLEMEN FROM PRD.

1030 HRS. GENTLEMEN FROM PRD ARRIVE AT OFFICE
HOWEVER, THEY WERE GIVEN ESCORT
REQUIRED BADGES, JEREMY TURNER
LEFT TO MAKE ARRANGEMENTS FOR
AN ESCORT.

1200 HRS. LUNCH

1300 HRS. ARRIVE AT FACILITY AND LEARNED
THAT BASELINE PLATING HAD BEGUN AT
1315 HRS. IN TANK 12-B. (4'x4' TANK
SPARTS WERE PLATED, HOWEVER, COUPONS
WERE NOT PLATED AT THIS TIME.

THE 5 PARTS WERE REVERSED FREQUENTLY FOR
2.5 MINUTES. THE SET POINTS FOR THE
PLATE WERE 3.6 VOLTS AND 133°F.

1400 HRS. MET WITH JEREMY TURNER. WE REQUEST
SAMPLE BOTTLES FOR TESTING, AN INDICATION
ON THE ANTICIPATED TIME THAT SAMPLES WOULD
BE FINISHED. HE TOLD US THAT THE PARTS
WOULD ONLY BE PLATED FOR 12 HRS. AND
THAT THEY WOULD BE PULSED AROUND 0130
OF 08-27-02. HE SAID THEY DIDN'T KNOW
THAT WE WANTED COUPONS ON BASELINE
TESTS SO THEY WERE NOT INCLUDED. WE
AGREED THAT THE COUPONS COULD BE
DONE IN ANOTHER TANK ON 08-27-02.
CONSIDERING THIS IS A BASELINE AND
THAT CHEMICAL MAKE UP IS SAME IN

3

MONDAY AUGUST 26, 2002 (CONTINUED)

1400 HRS (CONTINUED) FACIT TANK. JEREMY TURNER ALSO INFORMED US THAT CORPS CHRISTI ARMY DEPOT (CCAD) HAD ONLY SENT TWO SETS OF PARTS AND THAT A THIRD SET OF PARTS WOULD BE NEEDED TO PERFORM THE BASELINE, 2" OF PRD, AND 4" OF PRD. I THEN CALLED PETER Darcy of BENT LABORATORIES AND ASKED HIM TO CONTACT JEREMY SMITH OF CCAD ON SENTING AN ADDITIONAL SET OF PARTS.

1445 HRS WENT TO LAB ON AMMUNITION ARMY DEPOT BASE AND MET WITH STANLEY WILSON WHO SUPPLIED US WITH TESTING BOTTLES AND SYRINGES. I DISCUSSED WITH HIM HOW WE WERE TAKING SAMPLES AND THAT WHEN WE WERE THROUGH WE WOULD TURN SAMPLES OVER TO HIM. HE WILL SHIP THESE SAMPLES TO MARK FASER AT BENT LABORATORIES.

1530 HRS TOOK BASELINE SAMPLE OF CHROME SOLUT. FROM TANK 12-B.

1600 HRS MET JEREMY TURNER, AGREED TO RETURN TOMORROW MORNING AT 0800 HRS.

~~1830 HRS~~ MAY PHELPS INFORMED ME THAT HE ^HC

1630 HRS LEFT FACILITY FOR PAX.

1830 HRS MAY PHELPS OF PAXNL INFORMED ME THAT HE HAD A MESSAGE ON HIS HOTEL PHONE THAT SAID "NO PARTS AVAILABLE READ E-MAIL"

5

Tuesday August 27, 2002

0800 HRS. Arrive on Base. Met with Jeremy Turner. Jeremy informed us that coupons will not fit with parts in bath and that they may have to be cut in half. I immediately called Linda B. of Beyer Labs to find out what the minimum size requirements were for these test coupons. She stated that she would get back to me. We discussed the situation in field and decided that coupons can fit.

0830 HRS PRO fluid was added to tank, polypropylene balls had already been removed. Approximately 20 gallons were added to tank which created a level of 2.7". PRO technicians then removed approximately 5 gallons to bring level of fluid down to 2".

0845 HRS Pulled sample of Chrome Fluid N 200
Labeled sample (A)

2" PRO START

0845 HRS

08-27-02

0930 Parts were added.

CCAO - 114L2425-1

AAO - 12304148

AAO - 12286191

AAO - 12284387

1010 HRS Set Point 133°F

1 volt 210 Amps for 3 minute etch.

1013 HRS Plating Start

Set Point

3.6 volts 230 Amps 132°F

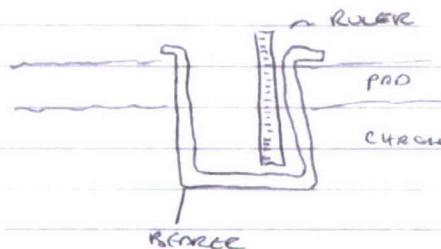
7

TUES AUGUST 27, 2002 (CONTINUED)

1611 HRS 3.9V 235A 132°F

THE TOP OF THE PRD FLUID WAS
DARK YELLOW IN COLOR

MAX PHELPS MEASURED THE DEPTH OF
PRD FLUID BY IMMERSING A BEAKER
INTO BATH AND READING THE LEVEL
OF FLUID LOOKING INSIDE BEAKER
SEE BELOW

1630 HRS PRD BLANKET WAS $2" \pm \frac{1}{8}"$ 1635 HRS SAMPLE OF CHROME TAKEN
~ 200 ml (B)

2140 HRS FLUID SURFACE WAS FOUND TO
BE A DARK YELLOW MAX MEASURED
THE PRD BLANKET TO BE $\sim 2" \pm \frac{1}{8}"$
POINTS SET AT 3.8V 236Amps 128°F

2215 HRS SAMPLE TAKEN FOR CHROME APPROXIMATE
200 ml (C)

2230 HRS PARTS REMOVED FROM BATH
ONE OF THE ANGLISTON PARTS
APPEARED TO HAVE A BAD PLATE
PART # 12304148

NOTE: VISUAL SPECULATION - THIS PART
HAS A LIP ON TOP. BELIEVE MAY BE
SOME PRD FLUID GOT TRAPPED WITHIN
LIP CAUSING BAD PLATE.

9

Tues. AUGUST 27, 2002 (CONTINUED)

2235 HRS DARK YELLOW DISAPPEARING, PRO FLUID BECOMING CLEAR.

Wed AUGUST 28, 2002

0600 ARRIVE ON BASE

0630 LOOKED AT TANK 12-13 - TOP SURFACE IS A BROWNISH-RED COLOR. DO NOT KNOW WHAT HAS CAUSED THIS.
TEMP. = $\sim 121^{\circ}\text{F}$

0645 HRS PRO TECHNICIAN ARRIVES TO ADD ADDITIONAL 2" BLANKET TO TANK BRINGING PRO BLANKET TO A TOTAL OF 4"

0650 HRS DECIDED TO TAKE SAMPLE OF PRO FLUID. SPECULATION THAT BROWNISH RED COLOR COULD BE RUST AND ASSOCIATED WITH USE OF IRON ANODES. A 200, SAMPLE WAS TAKEN WHILE PRO FLUID WAS BEING ADDED TO TANK. SAMPLE WAS LABELED

(01) 053802

0650 HRS
PRO - CURIOUS

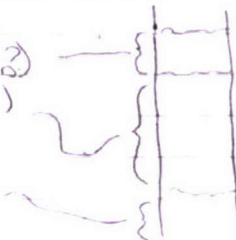
ANOMALY.

WHILE TAKING SAMPLE IT SHOULD ALSO BE NOTED COLOR CHARACTERISTICS

yellow (new PRO fluid?)

Red (PRO - Rust?)

Black (chrome?)



1

Wed. August 28, 2002 (CONTINUED)

0715 HRS RAKESH AND RAMESH OF PRD ARRIVE WITH DAN GRIESMER OF PORTER GUGERTINCO. DAN WAS A SELF-PROFESSED PLATING SPECIALIST AND WAS INVITED BY PRD TO EXAMINE TEST. THE BASELINE PARTS AND PARTS PLATED WITH 2" PRD HAVE BEEN LEFT OUT FOR VISUAL INSPECTION/CURIOSITY. DAN HAD PICKED UP ONE PART (12304148) AND HAD RUBBED IT, IN AN ATTEMPT TO CHIP IT WITH HIS FINGER NAIL BEFORE I COULD TELL HIM TO STOP. HE SAID THAT WHAT HE WAS DOING WOULD NOT AFFECT TESTING OF PARTS. I RESPONDED THAT "I DON'T CARE" AND HAD ASKED HIM NOT TO TOUCH ANY MORE PARTS.

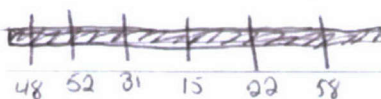
0800 HRS TOOK 200 ml sample (1) FOR START OF 4" PRD PLATE

0930 HRS PRD 4" PLATE BEGINS.
1 volt 180 amps 136°F FOR REVERSE ETCH

PLATING - 4 VOLTS 240 amps 136°F

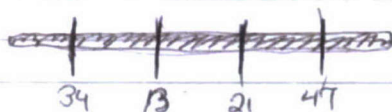
1030 HRS JERRY TURNER USING A OHMS TESTER? MEASURED IN AMPERAGE AT EACH ANODE CATHODE. RESULTS CAN BE SEEN BELOW.

CATHODE



pump

FRONT ANODE

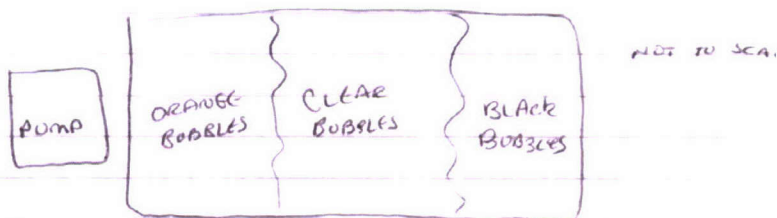


Wed. August 28, 2002

1430 HRS ARRIVE ON BASE FROM LATE LU

1505 HRS 4 VOLTS 250 AMPS 133°F
TANK IS AN ORANGE/YELLOW

NOTICED THAT BUBBLES BREAKING
SURFACE ARE DIFFERENT FROM EARLIER
BUBBLES ON LEFT SIDE WERE ORANGE
THAT WOULD CLEAR AWAY TO CLEAR BUBBLES
THAT BUBBLE WOULD BURST
BUBBLES IN MIDDLE CAME UP CLEAR
AND BURST BUBBLES ON RIGHT
SIDE CAME UP BLACK (CHROME)
BURST AND CHROME SLOWLY SAW
AGAIN. PLEASE SEE BELOW



1530 MAX PHELPS MEASURED THE RPD FLUID TO
BE 3 1/2". BELIEVE MAYBE THAT TURBULANCE
FROM PUMP IS MAKING IT HARD TO READ
SO MEASUREMENT WILL BE TAKEN AGAIN ~~LATER~~
WHEN PUMP IS SHUT DOWN
ALSO A 200 ML SAMPLE (E) WAS TAKEN AT
THIS TIME

2100 HRS TANK AND BUBBLING APPEAR TO BE SAME AS
CONDITION EARLIER IN DAY.

2120 HRS A 200 ML SAMPLE (F) WAS TAKEN.

2125 HRS PARTS PULLED - LOOK GOOD

2130 HRS PUMP TURNED OFF, RPD BLANKET MEASURED TO BE 3 3/4"

1E

Thursday August 29, 2002

0930 HRS EXAMINE PLATING BATH, PUMP HAS BEEN OFF FOR 12 HRS. PRO BATH IS NOT CLEAR. STILL APPEARS TO HAVE REDDISH BROWN COLOR, ALTHOUGH SAMPLE (C1) HAS SETTLED INTO TWO DISTINCT FLUIDS.

1000 HRS ALL 200 ml SAMPLES WERE TURNED OVER TO STANLEY WILSON (CHEMIST) OF ANNISTON Army Depot (256-235-7137) FOR SHIPMENT TO JOHN CAMMON AT BENET LABORATORIES WHERE SAMPLES WILL BE ANALYZED.

1030 HRS HAD DISCUSSION WITH PLATER, HE STATED THAT THEY WILL CLEAN CHROM TANK TWICE A YEAR AND REMOVE ~ 3-4" OF SLUDGE EACH TIME. THE OLD FLUID WAS REMOVED AND HE NOTICED THAT THE LEVEL OF CHROME SOLUTION HAD DROPPED ~ 2". WE ASKED THE PLATER/OPERATOR AND HE STATED THAT THIS WAS INDEED UNUSUAL.

1130 HRS HAD DISCUSSION WITH NICK DENNY. HE STATED THAT HE WOULD MAKE SERG PARTS WERE SHIPPED TO BENET LABS.

Thursday Sept 3, 2002

1300 HRS BEGAN WRITE UP OF TRIP REPORT

1500 HRS CALLED MARK FLESZAR AND LINDA BOHOCREEK OF BENET LABS. INFORMED THEM THAT EXPERIMENTS HAS BEEN COMPLETED AND THAT SAMPLES WILL BE SHIPPED SOON.

17

SEPT. 13 Friday

1400 HRS CHROME SAMPLES ARRIVED
FROM ANAD. ATTEMPTED TO
REACH MARK FLESCAR BUT HE
WAS NOT IN.

SEPT. 16 Monday

1000 HRS. RECEIVED TEST SPECIMENS FROM
ANAD

1400 HRS TURNED OVER TEST SPECIMENS
TO LINDA BOHACEK.

OCT. 21 Monday

0945 HRS RECEIVED RESULTS ON^C SAMPLE
ANALYSIS. TRANSFERRED RESULTS
TO ALICE CANYON, PHIL DORCY, AND
MARK FLESCAR.

DEC. 9 Monday

1200 HRS RECEIVED REPORT FROM MATERIALS
LAB. BEGAN FINAL REPORT FOR
PROJECT.

JUNE 2 Monday

0300 HRS LEAVE ALBANY

1100 HRS ARRIVE ANAD ACQUIRING BAGG

1130 HRS ACQUIRED SAMPLING EQUIPMENT FROM
ANAD CHEMIST

1230 HRS TONY DID NOT KNOW ABOUT PARTS TO BE
PLATED. HE SAID SPUR GEAR WAS TO
PREVIOUS ADAPT TO BE TESTED ON NOW.
WE WILL DEAR W/ THE ADAPTOR AND
LP SHAFT AND COUPONS

1530 HRS LEFT TO CHK IN AT HOTEL

19

JUNE 3 TUES.

0700 ARRIVE ON BASE w/ Jason Moore
of PNL.Tony Poulard informed us that
TANK 12-B'S RECTIFIER IS NOW
OPERATIONAL. THEY HAVE AN AD
ELECTRICIANS WORKING THE PROBLEM.0825 TOOK SAMPLE OF GOU ON PRD
FLUID FROM TANK 12-BSAMPLE XA - UNKNOWN MATERIAL
0825 HRSSAMPLE A CHARGE START OF PLATE
UNDER 4" PRD
0830 HRSSAMPLE B PRD - 4" PLATE START
0830 HRS0840 TOOK PICTURES OF PARTS/COUPONS AND
TANKS.ADAPTOR 12304148
LP SHAFT 12286191
3 COUPONS1030 HRS START PLATE
PRD APPEARANCE - YELLOWISH ORANGE

.070	.049	.065	.040	.010	.07
					.049
					.065
					.04
					.01
					<hr/>
					.294

2 min Reverse stroke @ 300 Amps.

TANK 12A SLOW, 12 min START PLATE 1100 HRS

21

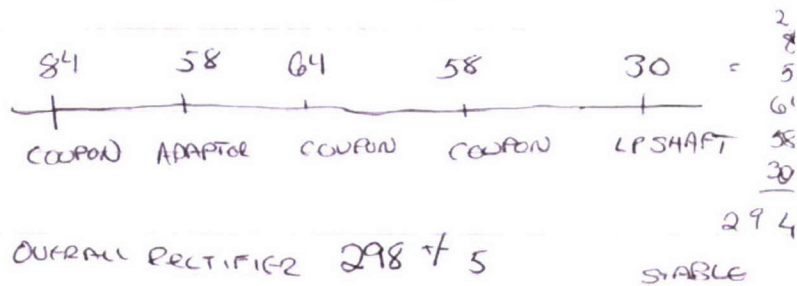
JUNE 3 TUES (CONTINUED)

1530 HRS BATH A LITTLE MORE ORANGE IN
COLOR BUT YELLOWISH ORANGE
OVER ALL

~~1645 HRS~~ JIC

1645 HRS SAMPLE C - CHROME
MID POINT IN PLATING CYCLE
WITH 4" PRO

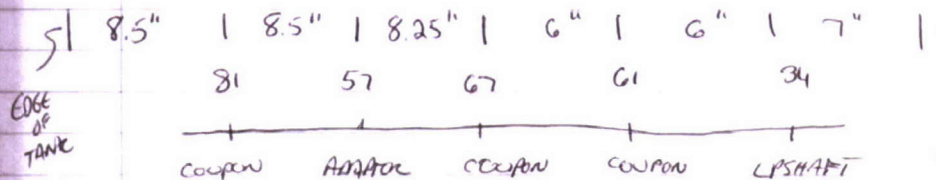
SAMPLE D - PRO
MID POINT IN PLATING CYCLE
WITH 4" PRO



2200 HRS BATH IS NOW ORANGE. - NO PRO LOSS

2215 SAMPLE E CHROME
END PLATING CYCLE w/ 4" PRO

SAMPLE F PRO
END PLATING CYCLE w/ 4" PRO



OVERALL RECTIFIER = 307 AMPS

81
57
67
61
34
<u>295</u>

295 AMPS

2:

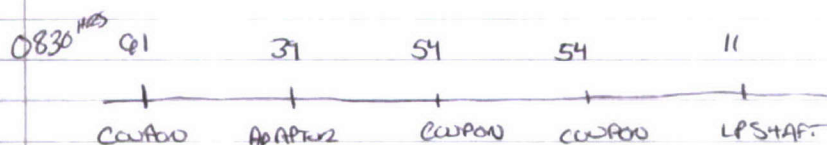
June 4 Wed.

0630 HRS - PRO APPEARS LEADISH BROWN
ART FROM PRO INCL IS REMOVING 2"
FROM TANK

0700 HRS TACKED TO PLATERS WE WILL BE
ABLE TO START PLATING IN ABOUT
1 HOUR

0740 HRS SAMPLE G - CITRUS
START PLATING CYCLE w/ 2" PRO

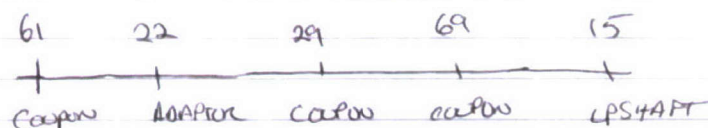
SAMPLE H - PRO
START PLATING CYCLE w/ 2" PRO



OVERALL RECTIFIER = 235 +/- 10

0915 AMPERAGE WILL NOT STABILIZE JUMPING
N 30 AMPS AT A TIME / 10 SECONDS
JUMPING IS ERRATIC AND NOT ON ANY
PERCEIVED CYCLE

1400 HRS YELLOWISH GRAY IN COLOR



RECTIFIER 210 +/- 10

25

June 4 Wed (CONTINUED)

1415 HRS Sample I Citron
Midpoint in 2" PRD PLATING CYCLE

Sample J. PRD
Midpoint in 2" PRD PLATING CYCLE

1430 HRS TALKED WITH Nick and Tony P.
NICK SAID HE WOULD DELIVER SAMPLES
(Liquo) TO Stanley Wilson on Friday
FOR SHIPMENT BACK HOME, Tony P.
SAID HE WOULD SHIP METAL SAMPLES TO
US AT BENT. HAVE MADE LABELS FOR
PARTS RUN @ 4" PRD, WILL ATTACH tomorrow
AFTER THEY HAVE BEEN BAKED AT 375° FOR
3 HRS.

1900 HRS BATH APPEARS Reddish Brown
AMPS ON RECTIFIER extremely LOW

1930 HR Sample K - CHROMING
END OF 2" PRD PLATING CYCLE

SAMPLE L - PRD
END OF 2" PRD PLATING CYCLE

1935 HRS AMPS ON RECTIFIER BACK UP AGAIN

35	25	28	35	9	3
+	+	+	+	+	3
Coupons	ADAPTOR	Coupons	Coupons	LP SHAFT	2
					2
					3

TOTAL Rectifier 157 amps 13

27

JUNE 5 THURS

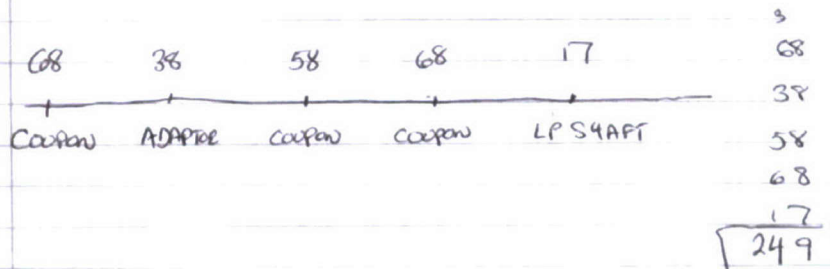
0630 HRS ARRIVE ON BASE - THERE STILL SEEMS TO BE ABOUT A $\frac{1}{4}$ INCH OF PRO ON TANKS

0700 HRS ADAPTOR IS HERE, PLATER IS PREPARING PARTS FOR PLATING, BAKING YESTERDAY'S.

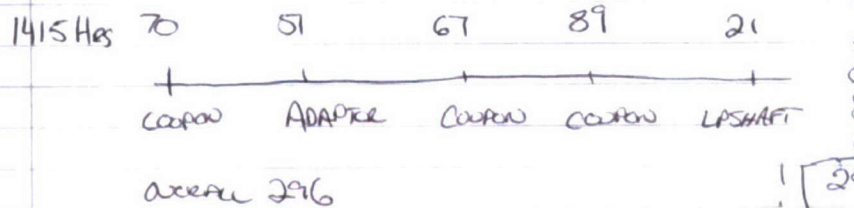
0800 START BASELINE PLATING

0800 SAMPLE M START OF BASELINE - CHEANE

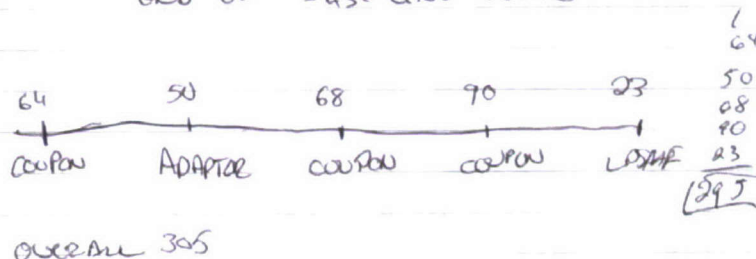
0830 OVERALL 245 \pm 5



1400 HRS SAMPLE N CHEANE
BASELINE PLATE MID POINT



1930 HRS SAMPLE O CHEANE
END OF BASELINE PLATING



29

June 12 2003

1445 HRS RECEIVED COMMUNICATION FROM TONY POLLARD AUTHORIZING USE OF PHOTOGRAPHS TAKEN DURING TEST.

July 2 2003

1300 HRS PLATED PARTS ARRIVED @ BENEF AND WERE TRANSFERRED TO MATERIALS GROUP FOR DESTRUCTIVE TESTING. TESTING WILL TAKE APPROXIMATELY ONE MONTH.

July 7 2003

0930 HRS CHEMICAL SAMPLES ARRIVED @ BENEF. TESTING CAN NOT COMMENCE UNTIL AFTER WWA SHUT DOWN, DUE TO INDUSTRIAL WASTEWATER TREATMENT PLANT BEING DOWN. TESTING WILL BEGIN FIRST OF AUGUST

August 12 2003

0830 HRS RECEIVED REPORT FROM MATERIALS GROUP.

August 20 2003

0900 HRS CONDUCTED MEETING WITH CHEM LAB ON DISPOSITION OF SAMPLES. APPARENTLY SAMPLES CONTAINING PRD HAVE "GUMMED" UP SOME OF THE MACHINES. THEY ARE GOING TO TRY A DIFFERENT APPROACH TO TESTING SAMPLES CONTAINING PRD.

31

SEPT. 4 2003

0630 HAS CITGM LAB SENT FINAL
RESULTS ON SAMPLES.
FINISHING FINAL REPORT.

Appendix G: PRD, Inc. Report Regarding Phase I Results

DATA ANALYSIS FOR PRD-EL1 DEMONSTRATION TEST PHASE I – PLATING QUALITY TESTING

Submitted To:

Dr. Steve W. Maloney
U.S. Army Engineer Research and
Development Center
Champaign, IL 61826-3482

By

Dr. Rakesh Govind
Director of Engineering, PRD Tech Inc.

&

Professor, Chem. Eng., University of Cincinnati

Tel: (859) 578-8010

Fax: (859) 578-8015

E-Mail: rgovind837@aol.com

February 2003

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 - (b) Adhesion Testing
 - (c) Quality Testing
 - (d) Porosity Testing
 - (e) Plating Hardness Test
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 - (a) Thickness Testing
 - (b) Adhesion Testing
 - (c) Quality Testing
 - (d) Porosity Testing
 - (e) Plating Hardness Test

Conclusion

References

Appendix 1 – Description of the Zero-Emission Process

ABSTRACT

PRD Tech, Inc. has developed a Zero-Emission process, which uses a proprietary liquid blanket, designed as PRD-EL1, which floats on the chromic acid liquid and reduces emission of chromic acid aerosols, emitted during hard chromium plating operation. Testing was conducted to evaluate the effect, if any, of the PRD-EL1 fluid on the quality of chromium plating. Testing was conducted at the Anniston Army Depot (ANAD), Anniston, AL. Plating was conducted on thirteen components and three test coupons (16 pieces total), supplied to Benét Labs by Anniston Army Depot (ANAD) and Corpus Cristi Army Depot (CCAD).

The majority of parts plated with PRD-EL1 fluid passed all qualitative (adhesion, quality, porosity) requirements. All plated parts passed the porosity test and the average hardness for all plated samples exceeded 700 HV and there were no individual hardness readings below 700 HV. Testing failures could be inadequate mixing, poor electrical contact in the cathode during plating, shadowing effect of the test coupons by the large adjacent parts during the plating operation, and surface contamination of the plating surface. After excluding the thickness and hardness data for the parts which were inadequately plated (plating thickness less than 0.01 inches), it was concluded that the average plating thickness and plating hardness, with and without the PRD-EL1 liquid blanket, did not exhibit any significant statistical differences, demonstrating that the PRD-EL1 liquid does not interfere with the plating process.

INTRODUCTION

Chromium plating of machinery parts produces a surface coating that helps reduce wear and corrosion. The military uses these processes as a cheap and effective way to combat the wear and corrosion of parts suffer during usage. The problem with chromium plating is the emission of a fine aerosol, during the plating process. Once in the atmosphere, the aerosol forms chromic acid. Hexavalent chromium has long been known to be a carcinogen and cause of perforated nasal passages, skin rashes and other medical problems. These emissions are controlled by capturing the aerosols at the surface with airflow above the vat, which enters an exhaust duct and exits from a stack outside the building.

PRD Tech, Inc. has developed a proprietary Zero-Emission process (Appendix 1) which uses an immiscible liquid that covers the top of the chromium bath during the plating process. This liquid, designated as PRD-EL1, prevents the aerosols of chromium from reaching the atmosphere by trapping the bubbles before they reach the liquid-air interface. If successful, this process may replace expensive scrubber technology currently used to deal with emissions.

Testing was conducted to evaluate the effect, if any, of the PRD Tech proprietary fluid on the quality of chromium plating. Testing was conducted in Building 114 on the Anniston Army Depot (ANAD), which houses the depot's metal finishing operations. Line 2 was used for the test, which had a total of four chromium vats. All test plating was conducted in vat 12B in Line 2.

The plating conditions used were as follows:

Voltage:	3 volts (Actual value is 1 V higher due to meter bias)
Current:	60 amps
Temperature:	128°F - 133°F (thermocouple)
Plating time:	12 hours
Reverse Etching:	Before plating at 3 volts, 65 amps for 2.5 minutes

Plating was conducted on thirteen components and three test coupons (16 pieces total), supplied to Benét Labs by Anniston Army Depot (ANAD) and Corpus Cristi Army Depot (CCAD). Two different layer thicknesses of the PRD Tech fluid were used: 2 inches and 4 inches. The baseline testing was conducted in accordance with

AMS 2406K ("Plating Chromium Hard Deposit"), para. 4.2.3) and AMS-QQ-C320 ("Chromium Plating"), para. 3.4.2 and 4.5.4).

The experimental design and components plated were as follows (Table G-1).

Table G-1. The experimental design and components plated.

TEST RUN	PART DESCRIPTION	PART ID
Test A Baseline (8/26/2002)	CCAD Part #1	114L2425-1
	CCAD Part #2	145H7359-1
	Adaptor Assembly	12304148
	Helical gear Shaft	12284387
	LP Comp. Shaft	12286191
Test Coupons were run on 8/27/2002 in a different vat.		
Test B 2" PRD Fluid (8/27/2002)	CCAD Part #1	114L245-1
	Adaptor Assembly	12304148
	LP Comp. Shaft	12286191
	Helical Gear Shaft	12284387
	Test Coupons	
Test C 4" PRD Fluid (8/28/2002)	CCAD Part #2	145H7359-1
	Adaptor Assembly	12304148
	LP Comp. Shaft	12286191
	Helical Gear Shaft	12284387
	Test Coupons	

PLATING TESTS CONDUCTED

The following tests were conducted to evaluate the quality of plating:

- I. Qualitative: Visual observations of the plated parts before thermal annealing
- II. Quantitative:
 - (a) Thickness Test in accordance with ASTM B487
 - (b) Adhesion Test in accordance with ASTM B571 (para. 8)
 - (c) Quality Test in accordance with AMS 2406K (para. 3.5)
 - (d) Porosity Test in accordance with AMS-QQ-C-320
 - (e) Hardness Test in accordance with ASTM B 578

RESULTS AND DISCUSSION

1. Test Run A – Baseline Test

Visual inspection of the plated parts indicated that the overall plating quality was very good. Small amounts of noncritical bits resulting partly from the gas bubble and partly from the amount of plating. Plating thickness varied in the range of 0.006 to 0.01 inches.

(a) Thickness Testing

Thickness tests were conducted by obtaining multiple cross-sections from each component/coupon. These cross-sections were mounted, polished, and etched with a 2 percent nital solution. Component 145H7359-1 (CCAD Part # 2) did not etch with nital. An X-Ray fluorescent chemical analysis was performed on the base metal of this piece and it was qualitatively identified as a stainless steel. All other components were successfully etched, indicating the base material was a steel alloy. Results of thickness measurements are given in Table G-2 and shown as Figure G-1.

Table G-2. Results of the thickness measurements for the Baseline Test.

Part No.	Baseline Thickness (in.)
114L2425-1	0.0113
145H7359-1	0.0137
12304148	0.0074
12286191	0.0232
12284387	0.0149
Coupon	0.0038

It can be seen that the plating thickness for the coupon was significantly less than for all other parts. One reason for this lower plating thickness may be that the coupon were plated separately in a different vat on the second day (8/27/2002), since they were unavailable until the second day of the demonstration test run. Since vat 12B was being used for Test Run B on the second day, these coupons were plated in one of the vats located across in Line A. Since the condition of plating in this vat may have been different (different concentration of chromium, temperature, current, voltage, etc.), the plating thickness cannot be directly compared with the parts plated in vat 12B at the same time.

The placement of the parts in the tank is shown below Figure G-2.

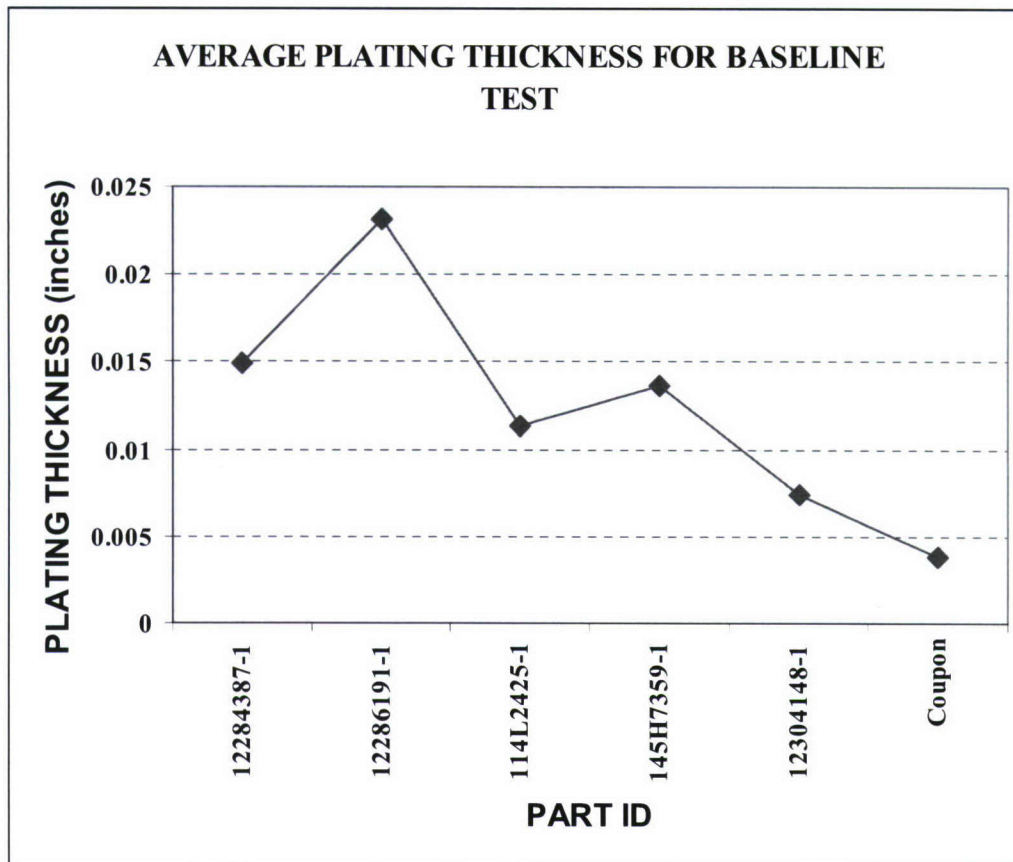


Figure G-1. Plot of plating thickness of each piece for Baseline Test.

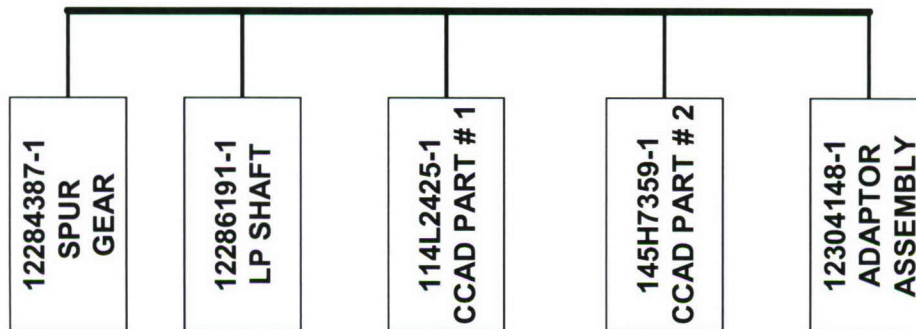


Figure G-2. Placement of the parts in the tank.

The LP Shaft (Part ID 12286191-1) had the maximum plating thickness (0.0232 in.) while the Adaptor Assembly (Part ID 123041481-1) had the minimum plating thickness of 0.0074 in. (not including the coupon, since it was plated in a different vat). This may be due to a wall effect of the tank since the Adaptor Assembly was at one end while the LP Shaft was more in the center of the tank. It is well known that tank mixing is less near the walls of the tank and more in the center of the tank, especially for deep tanks, as in this case. The Spur Gear (Part ID 12284387-1) had a plating thickness in between the highest and lowest values, even though it was at

the other end of the tank. The part's geometry also plays an important part. The Spur Gear was a long part, with the section being plated located at the bottom section of the part, while the Adaptor Assembly was a shorter part with plating section located near the top portion of the part. Since liquid mixing is from the bottom of the tank (spargers located at the bottom of the tank), parts with plating section located at the bottom will receive a thicker plating than parts with plating section located near the top of the part.

An example of a cross-section used for thickness measurements can be seen in Figure G-3.

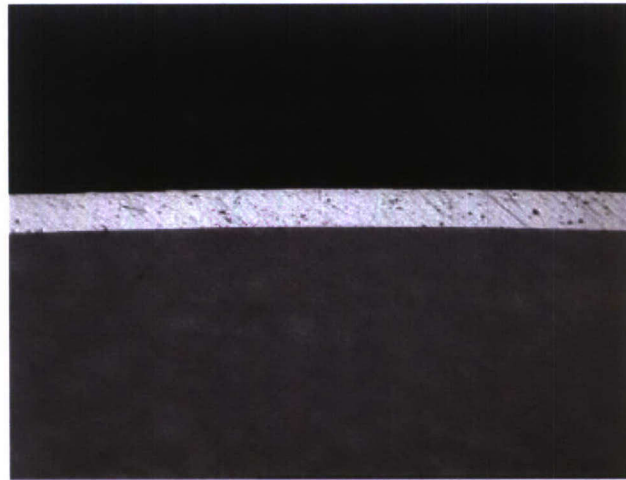


Figure G-3. Typical view of chromium plating sample used for thickness measurement.

(b) Adhesion Testing

Adhesion testing was conducted in accordance with ASTM B571 (para. 8). The Grind Saw Test was performed on eight areas for each specimen, and the areas were then examined at 5X magnification. Satisfactory adhesion was observed in all of the specimens except 145H7359-1 Baseline. Failure in the adhesion test indicates that the surface prior to plating was not cleaned completely or during the placement of the part in the vat, the surface was touched or contaminated by some impurity. It should be noted that this was the same part that did not etch with 2 percent solution of nital, since it was made of stainless steel while all other materials were made of a steel alloy. The base metal also plays a critical part in adhesion of the chromium plating, and ASTM B-254 entitled "Practice for Preparation of and Electroplating on Stainless Steel" should provide all the answers to this issue.

Representative photographs of a good adhesion sample (a) and the failed sample (b) are displayed in Figures G-4 and G-5. Figure G-4 is an example of a sample that passed adhesion criteria and Figure G-5 is an example of Sample 145H359-1 Baseline that failed the adhesion test due to the lifting/peeling of the chromium plating.

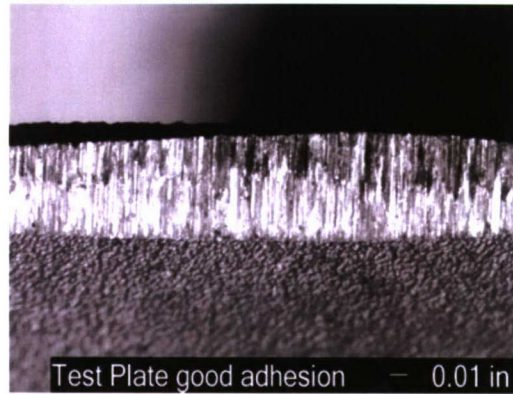


Figure G-4. Sample that passed adhesion criteria.

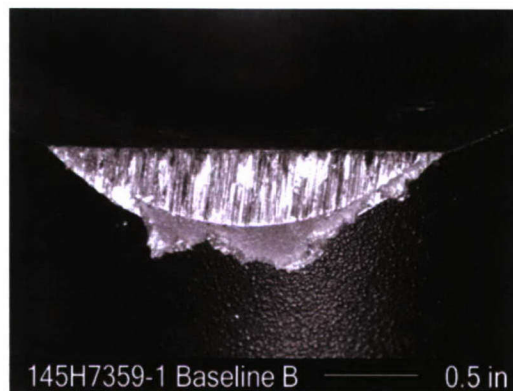


Figure G-5. Sample 145H359-1 Baseline that failed the adhesion test.

(c) Quality Testing

Quality testing was performed on each piece (components, coupons) by visual inspection in accordance with AMS 2406K (para.3.5). The specification requires that the plating be firmly bonded to the base metal, while maintaining smooth and uniform appearance. Pinholes and other small imperfections that resulted from the plating not filling in defects on the surface metal are considered acceptable. A magnification of 5X was used to inspect the pieces.

All the pieces plated in the baseline test passed this quality test.

(d) Porosity Testing

Porosity testing was conducted in accordance with AMS-QQ-C-320. A ferroxyl test was completed by soaking filter paper in ferroxyl solution and heating to 180-200 degrees F. The paper was then placed over the entire plated area on each piece for ten minutes. Blue markings occurred on the filter paper as a result of the reaction of the base metal with the solution. These markings are indicative of porosity.

All the pieces plated in the baseline test passed the porosity test.

(e) Plating Hardness Test

Hardness testing was completed in accordance with ASTM B578. A Vicker's indenter with a 100 gram load was used. The minimum acceptable hardness as per the standard was 700 HV. The average hardness for all samples was above the 700 HV minimum (see Table G-3). Additionally, there were no individual hardness readings below 700 HV.

Table G-3. Results of plating hardness test for the Baseline pieces.

Part Number	Baseline Hardness (HV)
114L2425-1	906.9
145H7359-1	921.33
12304148	855.33
12286191	877.07
12284387	855.33
Coupon	844.87

Figure G-6 shows a graph of the Hardness Test results. The test coupons had the lowest hardness and again this can be attributed to the fact that these coupons were plated in a separate vat, wherein the plating conditions may have been different than in vat 12B where the rest of the pieces were plated. Part 145H7359-1 had the highest hardness (921.33 HV), and since this part was stainless steel, the stainless steel base metal may have played a part in getting this higher number on the hardness.

The Adaptor Assembly (Part ID 12304148-1) had the lowest hardness number and this part was located at one end of the bath, as discussed before in the plating thickness section of this report (refer to Section 1a).

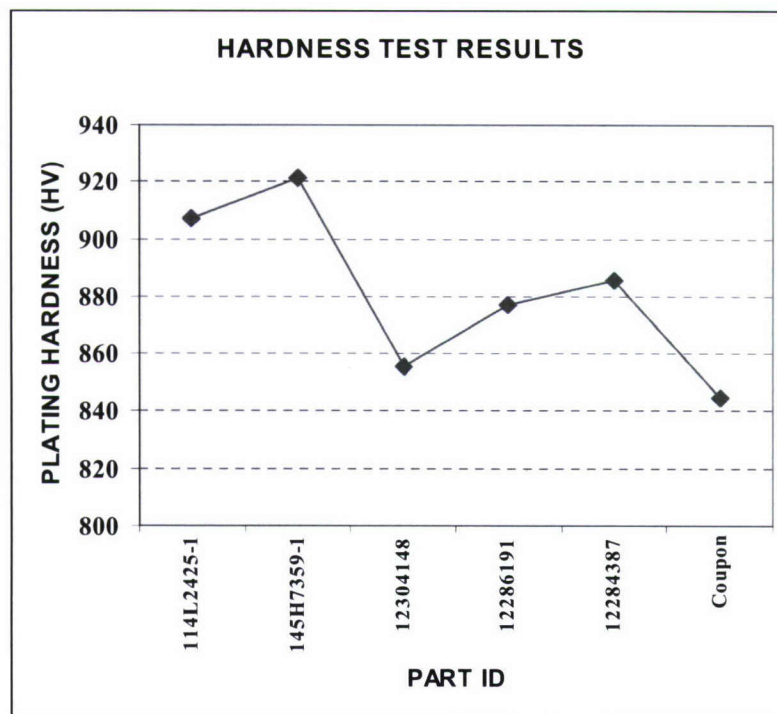


Figure G-6. Results of the hardness test for the Baseline pieces.

2. Test Run B – 2" PRD-EL1 Fluid Layer Thickness

The location of the parts plated was as shown below (Figure G-7).

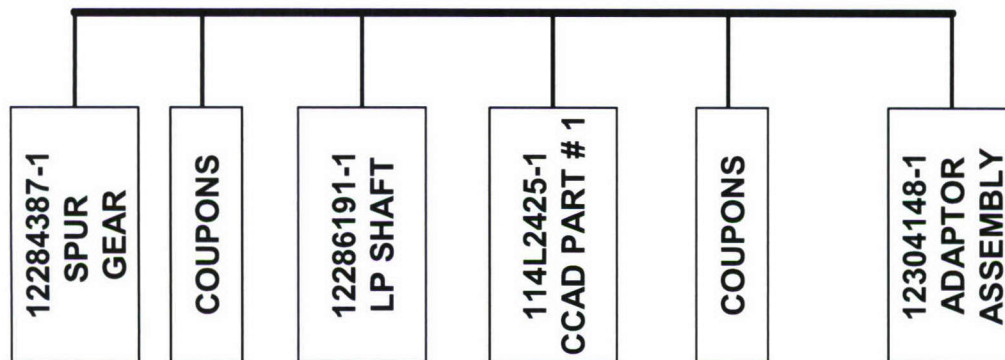


Figure G-7. Location of the parts plated.

The following qualitative observations were made for the plated parts:

Adaptor Assembly (Part ID 12304148-1)

Noticeable discoloration along the side of plated part. It seemed that the plating was much less in this area. Possible reasons include: (1) Wax didn't get removed

completely during heat and reverse etching performed prior to beginning of the forward plating operation; (2) High contaminants of bath by ferrous oxide (visible rust). Use of Fe anodes could have resulted in the deposition of rust particles in this area due to slight magnetization of the surface, generally due to machining operation; (3) Iron coupled with presence of molten wax could have temporarily blanketed this area; (4) Operation of the pump which was just started prior to this test run stirred up sediments which may have affected the quality of plating, i.e., no plating in those areas; (5) Possible interruption (poor contact) in the electrical contact of the cathode bar holding the parts, due to PRD-EL1 drip on the bar surface during the height measurement. Height of PRD-EL1 liquid layer was measured prior to beginning the test run by inserting the ruler and tape through the PRD-EL1 liquid in the general surface of the vat near the cathode bar. In addition, this part was located at one end of the tank, where lack of liquid mixing due to tank wall may have impacted the quality of plating.

Spur Gear (Part ID 12284387-1)

Very rough plating was observed on the surface.

LP Shaft (Part ID 12286191)

Plating comparable to Baseline Test, Test Run A was obtained. Suspect some blistering due to bath contamination.

CCAD PART # 1 (Part ID 114L2425)

Plating comparable to Baseline Test, Test Run A was obtained.

Test Coupons

Major “trees”, needle like structures were found along the edges. It seems that the edges were plated preferentially than the surface. Surface roughness was also noticeable as in other parts, indicative of bath contamination enhanced by the operation of the pump and sparger unit.

Results of quantitative testing are given below.

(a) Thickness Testing

Thickness tests were conducted by obtaining multiple cross-sections from each component/coupon. These cross-sections were mounted, polished, and etched with a 2 percent nital solution. Component 145H7359-1 (CCAD Part # 2) did not etch with nital. An X-Ray fluorescent chemical analysis was performed on the base metal of this piece and it was qualitatively identified as a stainless steel. All other components were successfully etched, indicating the base material was a steel alloy. Results of thickness measurements are given in Table G-4 and shown in Figure G-8.

Table G-4. Results of thickness measurements for pieces plated with 2" PRD-EL1 fluid.

Part No.	2" PRD Thickness (in.)
114L2425-1	0.004
145H7359-1	
12304148	0.007
12286191	0.0131
12284387	0.0097
Coupon	0.0031

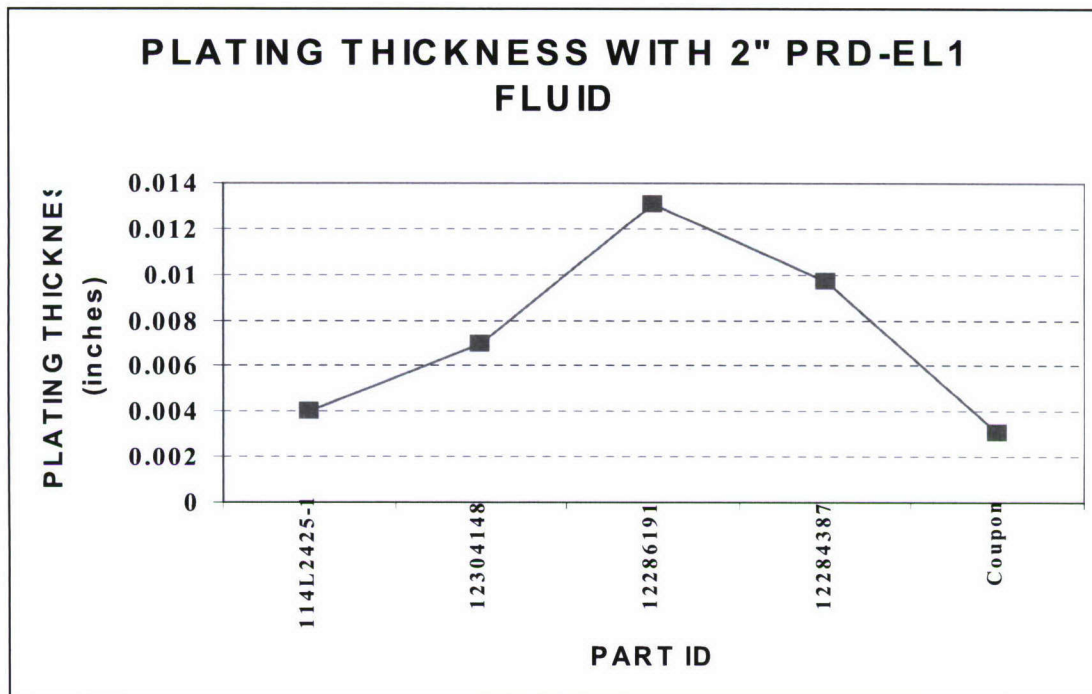


Figure G-8. Plot of plating thickness for pieces plated with 3" PRD-EL1 fluid.

The LP Shaft (Part ID 12286191) had the highest plating thickness (0.0131 inches) and the CCAD Part # 1 (Part ID 114L2425-1) had the minimum thickness (0.004 inches), not including the test coupons. The test coupons had the overall minimum thickness and this is mainly because of the location of the coupons within the tank. The coupons were located between two parts, and this essentially caused the small coupon to fall into the “plating shadow” of the adjacent parts, which meant that the adjacent parts got plated preferentially to the coupon. However, due to higher current density (amperes/area) on the coupon edges, preferential plating occurred on the edges, resulting in formation of trees and needle-like structures.

Also, as in the previous test, the LP Shaft was placed at or near the center of the tank, which may explain its maximum plating thickness and good quality of plating. The CCAD Part # 1 (Part ID 114L2425-1) had good plating quality but the plating thickness was much less than the LP Shaft. This may have been due to poor electrical contact, and this was demonstrated experimentally when the current flow was measured at each cathode. The data on current flows, as measured during the plating test, is shown below (Figure G-9).

The CCAD Part # 1 had almost 1/3 of the current flow as compared to the other parts, and this explains why the plating thickness is also minimum. This shows that poor contact can lead to low plating thicknesses, as is well known. In comparison, the LP Shaft had almost twice the current flow when compared to the CCAD Part # 1. It should be noted that the current flow is also proportional to the area being plated, and although data on plated areas is not available, from visual inspection it did not seem that the CCAD Part # 1 had significantly less plating area than the LP Shaft. This needs to be verified using the actual plating areas, which can be easily determined from the plated parts.

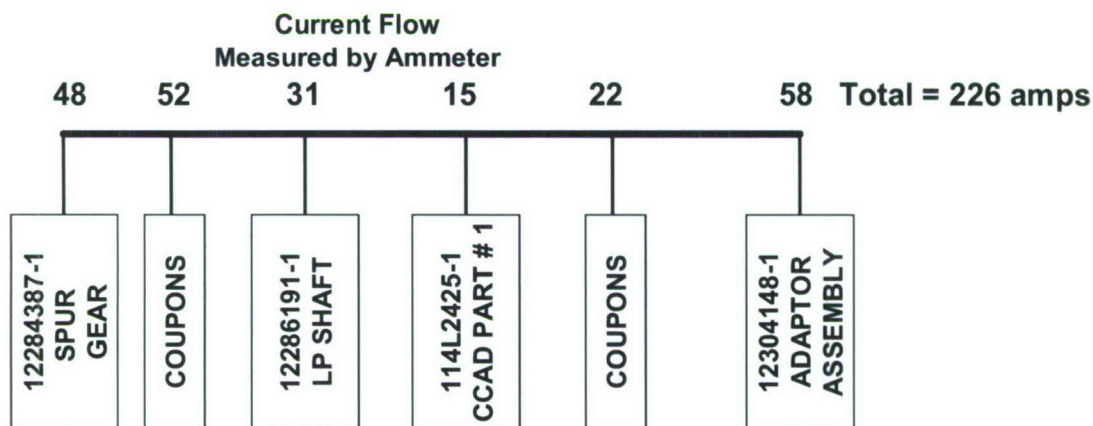


Figure G-9. The data on current flows.

(b) Adhesion Testing

Adhesion testing was conducted in accordance with ASTM B571 (para. 8). The Grind Saw Test was preformed on eight areas for each specimen, and the areas were then examined at 5X magnification. Satisfactory adhesion was observed in all pieces except the 2" coupon, which failed the test. The photograph below in Figure G-10 shows the failed coupon sample.

As discussed under the Plating Thickness test, the 2" coupon was not plated satisfactorily mainly due to "shadowing" by the adjacent parts, and this explains why it also failed the adhesion test.

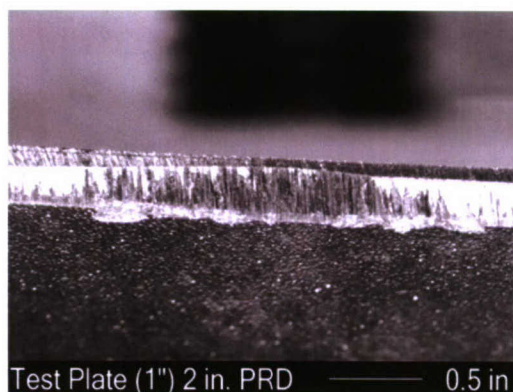


Figure G-10. Photograph of the 2" Coupon that failed the adhesion test.

(c) Quality Testing

Quality testing was performed on each piece (components, coupons) by visual inspection in accordance with AMS 2406K (para. 3.5). The specification requires that the plating be firmly bonded to the base metal, while maintaining smooth and uniform appearance. Pinholes and other small imperfections that resulted from the plating not filling in defects on the surface metal are considered acceptable. A magnification of 5X was used to inspect the pieces.

All pieces passed the test except the Adaptor Assembly (Part ID 12304148-1), which exhibited a burst bubble in the plating surface (Figure G-11).



Figure G-11. Ruptured bubble in Part ID 12304148-1.

(d) Porosity Testing

Porosity testing was conducted in accordance with AMS-QQ-C-320. A ferroxyl test was completed by soaking filter paper in ferroxyl solution and heating to 180–200 °F. The paper was then placed over the entire plated area on each piece for 10 minutes. Blue markings occurred on the filter paper as a result of the reaction of the base metal with the solution. These markings are indicative of porosity. All the pieces plated in the baseline test passed the porosity test.

(e) Plating Hardness Test

Hardness testing was completed in accordance with ASTM B578. A Vicker's indenter with a 100 gram load was used. The minimum acceptable hardness as per the standard was 700 HV. The average hardness for all samples was above the 700 HV minimum (see Figure G-12). Additionally, there were no individual hardness readings below 700 HV.

The highest hardness was measured in the LP Shaft (Part ID 12286191-1) and the minimum hardness was in the CCAD Part # 1 (Part ID 114L2425). The hardness plot mimics the plating thickness plot, shown in Figure G-8. So the discussion presented earlier for the plating thickness also applies to the hardness data, although the differences in the hardness numbers are not large. It is important to point out that since all the pieces passed the porosity test, the hardness should be above the minimum value of 700 HV. Also, differences in current flow, as noted earlier, could have contributed to less plating and lower hardness numbers for the CCAD Part # 1 (Part ID 114L2425).

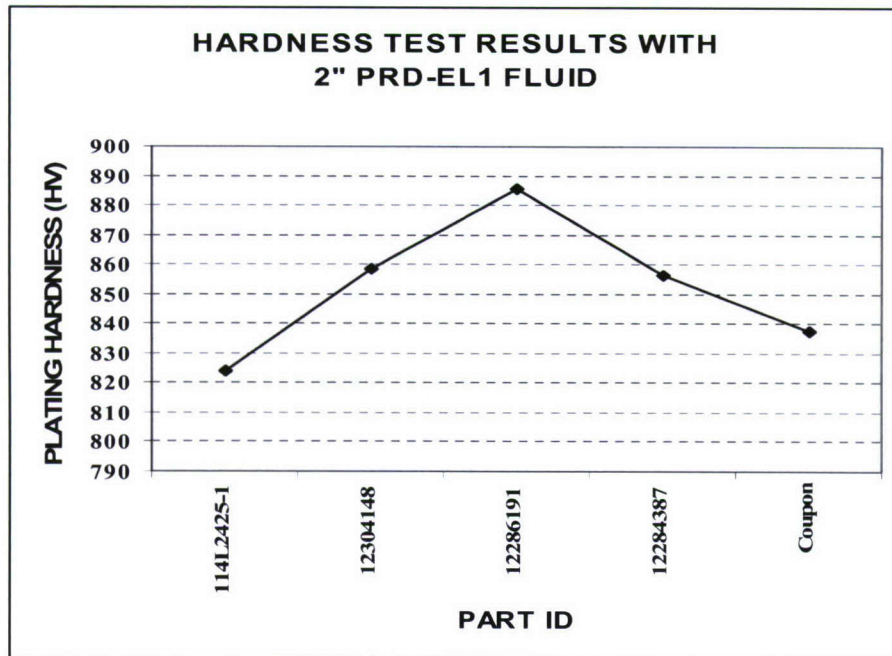


Figure G-12. Results of the hardness test for pieces plated with 2" PRD-EL1 fluid.

3. Test Run C – 4" PRD-EL1 Fluid Layer Thickness

The location of the parts within the bath are shown below (Figure G-13).

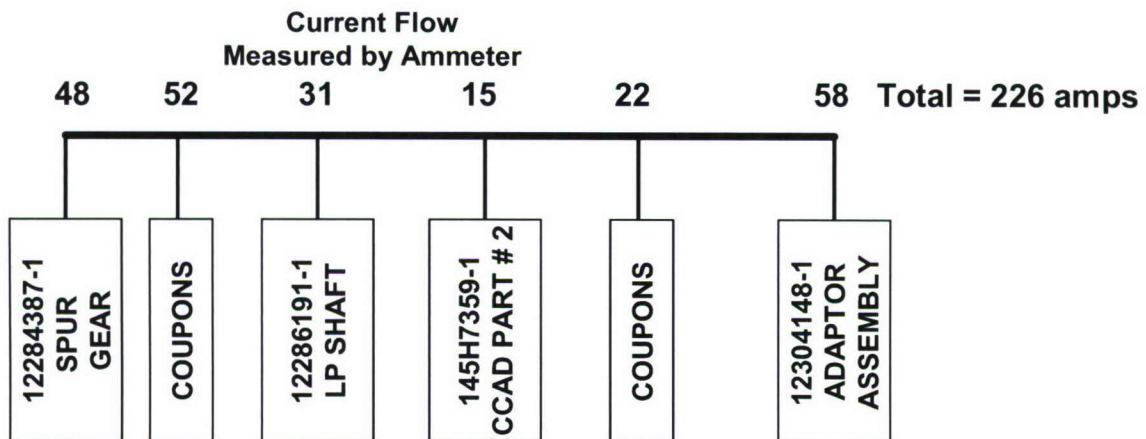


Figure G-13. Location of parts within the bath.

It seemed that, overall, there were no visibly evident signs of any possible plating quality issues, unlike the Test Run B, where some discolorations, rough plating, blistering, etc. were observed.

It was also noted that particles of iron oxide and others, present in the chromic acid bath due to prior reverse etching, were not present in substantial concentration in the PRD-EL1 liquid layer (2 inches thick) after the 2" plating test (current flow switched off, mixing pump running), which gave the PRD-EL1 layer an almost clear appearance visually. However, before the 4" test was begun next day, and before new liquid was added to increase the liquid layer thickness from 2" to 4", with the mixing pump running overnight, it was observed that the PRD-EL1 layer (2" thick) had a higher concentration of iron oxide and other particles, than were present the day before after completion of the 2" test.

When plating is conducted, bubbles of hydrogen and oxygen gases rise through the chromic acid, and then either break into smaller bubbles (microbubbles) as the gas enters the PRD-EL1 layer at the chromic acid-PRD-EL1 interface (when the rise velocity of the gas bubbles is higher and the bubbles are bigger), causing the PRD-EL1 Layer to have a whitish appearance in some places during plating, or bubble without breakage through the PRD-EL1 layer. After plating has been concluded, and after the plating current has been switched off, the gas microbubbles remain suspended in the PRD-EL1 layer for some time, and slowly rise to the surface of the PRD-EL1 layer. However, due to the presence of these microbubbles, the average density of the PRD-EL1 layer is reduced, since the gas bubbles have a significantly lower density than the PRD-EL1 liquid itself. Due to this lower average density of the PRD-EL1 layer with gas microbubbles, the solid particles of iron oxide and others that enter the liquid layer due to mixing, are unable to stay in the PRD-EL1 liquid layer and settle at a much faster rate, due to a higher density difference. This gives the liquid layer a clearer appearance.

However, after plating has been concluded, and after sufficient time has elapsed (overnight) to allow the microbubbles to escape into the ambient air, with the mixing pump running, the solid particles are able to enter and stay in the liquid layer longer, due to lower density difference, thereby giving it a murkier appearance visually.

It was also observed that after plating with 4" thickness of PRD-EL1 layer, the level in the plating tank had fallen significantly more than when plating was conducted with 2" layer, or even when no PRD-EL1 layer was present (base case, with air running).

With PRD-EL1 layer as a blanket with no on-going plating (no gas bubbling), evaporation rate of water through the liquid blanket is reduced. Laboratory measurements, conducted at 130 degrees F, with no liquid agitation of chromic acid or liquid

blanket, experimentally showed that water evaporation rates were 0.01 – 0.03 gallon/ft².hr using a 2" thick layer of PRD-EL1, and were 0.004 – 0.013 gallon/ft².hr for a 4" thick layer of PRD-EL1. The reason for the variability was due to the fact that humidity of ambient air above the blanket was not controlled when these measurements of evaporation rate were made.

When plating is conducted with no PRD-EL1 blanket, hydrogen and oxygen gas bubbles, formed due to electrolysis of water, rise through the chromic acid and carry water vapor. This causes water loss to increase during plating than when no plating is being conducted. The amount of gas bubbles generated during plating depends on the amperage and voltage at which the plating is conducted, i.e., current density.

In the laboratory, we have observed higher water loss with increasing PRD-EL1 blanket thickness, during gas bubbling. The evaporation rate of water at 130 degrees F for 2" PRD-EL1 blanket thickness increased by 10 percent when compared with the same conditions (temperature, gas flowrate through the chromic acid) with no PRD-EL1 blanket and by 17 percent with 4" PRD-EL1 blanket layer thickness. These results are expected to change with changes in gas flowrate (amperage and voltage of plating), plating temperatures, etc. This increase is attributed to several factors, discussed earlier: (1) increased water vapor content inside the gas bubbles, as they pass from the chromic acid liquid to the liquid blanket layer; (2) spouting of chromic acid as the gas bubbles, especially large size, move through the blanket layer, which causes the chromic acid to spread on the blanket liquid's surface, increasing evaporation rate of water; (3) increased tendency for gas to accumulate between the chromic acid-PRD-EL1 interface, resulting in periodic bursting, accompanied by spouting effect.

It should be noted that increased water loss during plating, and reduced water loss when no plating is being conducted, is actually beneficial for application of PRD-EL1 liquid blanket technology for electroplating tanks. When no liquid blanket is used, evaporation of water, especially during warm weather conditions, requires regular additions of water, and usually the water from the rinse tanks is used for this purpose. This allows electroplating shops to operate with no net wastewater (chromium present) discharge. Results of quantitative testing are given below.

(a) Thickness Testing

Thickness tests were conducted by obtaining multiple cross-sections from each component/coupon. These cross-sections were mounted, polished, and etched with a 2

percent citric acid solution. Component 145H7359-1 (CCAD Part # 2) did not etch with citric acid. An X-Ray fluorescent chemical analysis was performed on the base metal of this piece and it was qualitatively identified as a stainless steel. All other components were successfully etched, indicating the base material was a steel alloy. Results of thickness measurements are given in Table G-5 and shown in Figure G-14.

Table G-5. Results of plating thickness measurements for 4" PRD-EL1 liquid.

Part No.	4" PRD Thickness (in.)
114L2425-1	
145H7359-1	0.0126
12304148	0.0094
12286191	0.0129
12284387	0.0183
Coupon	0.006

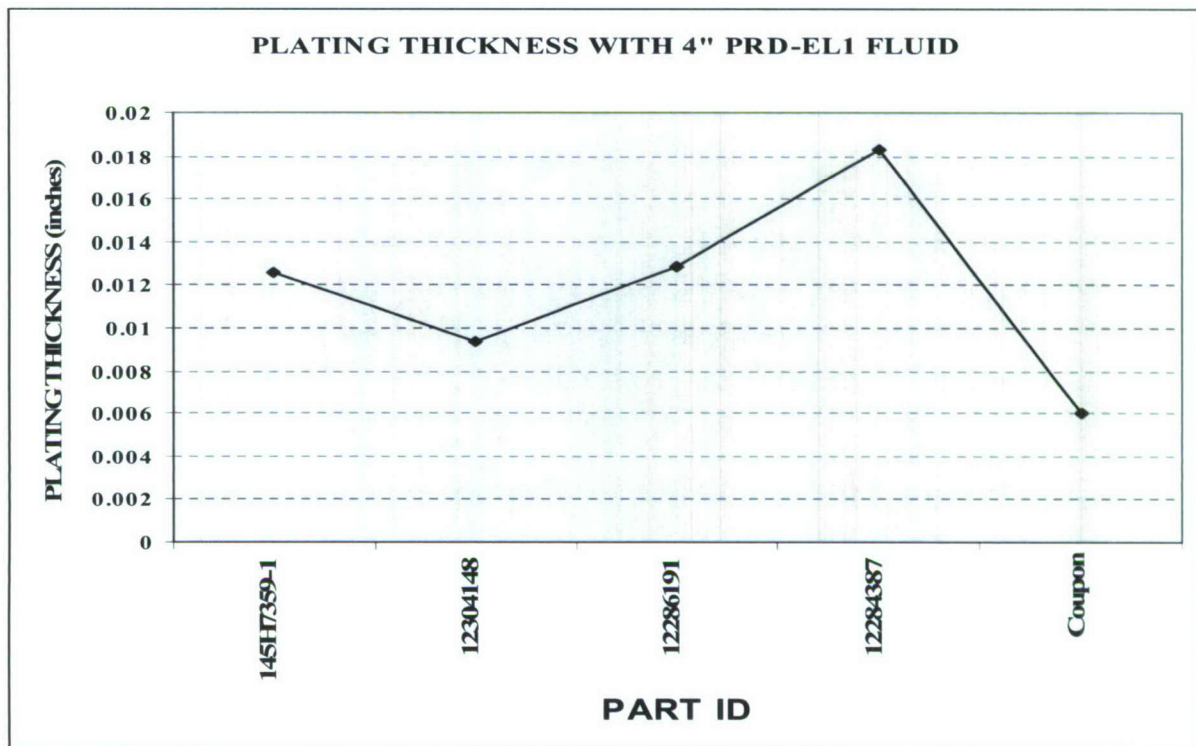


Figure G-14. Plot of plating thickness with 4" PRD-EL1 fluid.

The Spur Gear (Part ID 12284387) had the highest plating thickness, while the Adaptor Assembly (Part ID 12304148) had the lowest plating thickness. For both these cases, the parts were located at the two ends of the tank and current measurements at these two cathode showed comparable current flows (refer to figure at the beginning of this section, which shows the placement of the parts). The test

coupon had the overall lowest plating thickness. While the lack of plating in the test coupon case can be explained by the “shadowing” effect, discussed earlier, differences in plating thicknesses between the Spur Gear and the Adaptor Assembly are more difficult to explain. Perhaps these differences are not significant, which can be determined if the raw data on thickness measurements is analyzed to determine the standard deviations, rather than just the averages, as in the plot above.

It should be noted that all the pieces plated to greater thicknesses in this run than in the previous run (Run B with 2” PRD Fluid), since the greatest plating thickness in Run B was 0.0131 inches whereas in this Run it was 0.0183 inches. This can be attributed to the fact that the voltage, amperage and temperature conditions in Runs B and C were as follows:

EXPERIMENTAL RUN PLATING CONDITIONS

Run B - 2” PRD-EL1 Fluid:

Voltage 3.6 volts

Current 220 amperes

Temperature 132° F

Run C - 4” PRD-EL1 Fluid:

Voltage 4 volts

Current 240 amperes

Temperature 136° F

Even if the voltage and current effects are neglected, the higher temperature in Run C must have contributed to greater plating rates, resulting in greater plating thicknesses for all parts. Figure G-15 shows the comparison of plating thicknesses for Runs B and C.

As can be seen, plating thicknesses with 2” PRD-EL1 Fluid were consistently lower than with 4” PRD-EL1 fluid layer thickness, which demonstrates that the plating rates with 2” PRD-EL1 fluid layer were lower than with 4” PRD-EL1 layer, and this decrease is due to lower voltages, current and bath temperature.

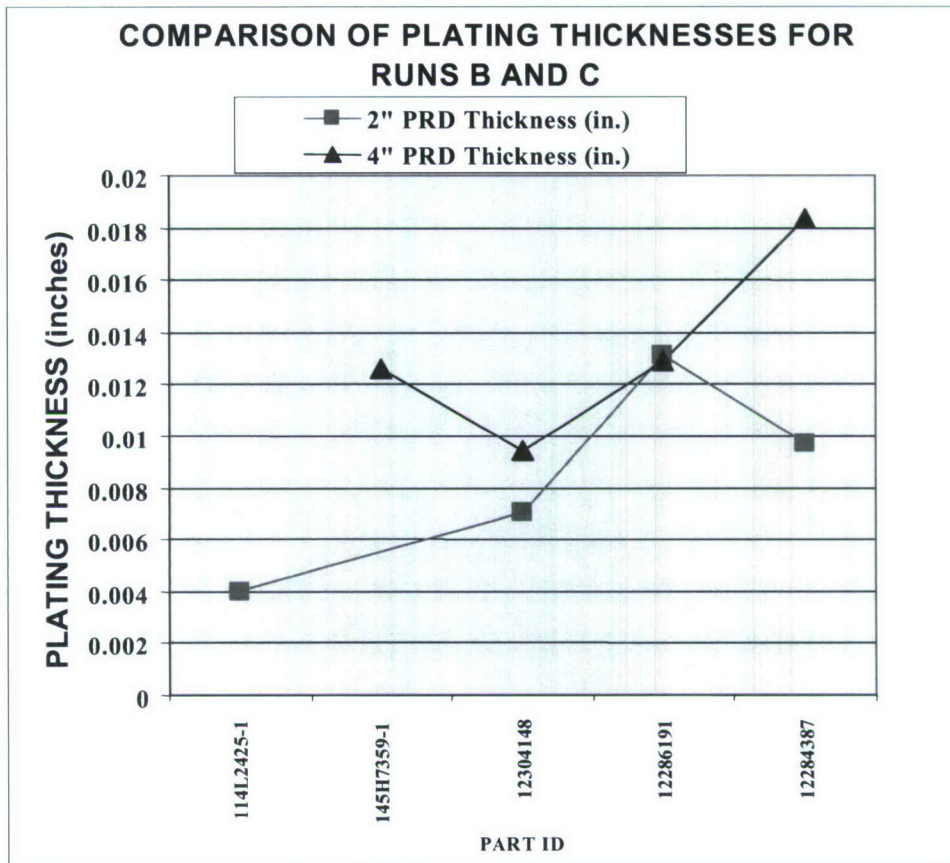


Figure G-15. Comparison of plating thickness for Runs B and C.

(b) Adhesion Testing

Adhesion testing was conducted in accordance with ASTM B571 (para. 8). The Grind Saw Test was performed on eight areas for each specimen, and the areas were then examined at 5X magnification. Satisfactory adhesion was observed in all pieces except the 4" coupon, which failed the test. Figure G-16 shows a photograph of the failed 4" Coupon.

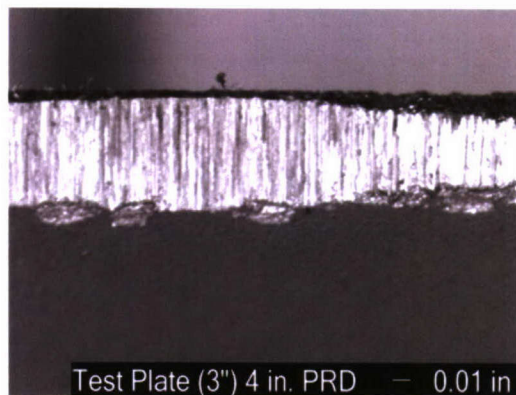


Figure G-16. Photograph of the 4" Coupon which failed the adhesion test.

The 4" coupon failure can be attributed to poor plating due to the "shadowing" effect, which has been discussed before in previous sections.

(c) Quality Testing

Quality testing was performed on each piece (components, coupons) by visual inspection in accordance with AMS 2406K (para. 3.5). The specification requires that the plating be firmly bonded to the base metal, while maintaining smooth and uniform appearance. Pinholes and other small imperfections that resulted from the plating not filling in defects on the surface metal are considered acceptable. A magnification of 5X was used to inspect the pieces.

All pieces passed the test, except the Spur Gear (Part ID12284387). It exhibited a burst "bubble" in the plating surface (see Figure G-17).

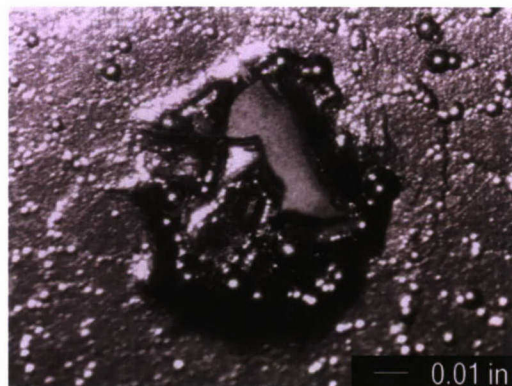


Figure G-17. Photograph of a ruptured bubble in sample ID 12284387-1.

It should be noted that this sample also had the maximum plating thickness and was physically located at one end of the tank. The previous time such a failure had

occurred was in the previous run (Run B with 2" PRD-EL1 liquid) and in that case (Adaptor Assembly, Part ID 12304148-1) the piece was also located at one end of the tank, rather than in the middle. Gas bubbles stick to the plating surface when there is insufficient liquid mixing around the part and this lack of mixing predominates near the tank walls. This type of plating failure is mainly due to lack of liquid movement which allows hydrogen/oxygen gas bubble to stick to the part's surface during plating and prevent plating in the area covered by the gas bubble. Eventually the gas bubble bursts creating the failure.

(d) Porosity Testing

Porosity testing was conducted in accordance with AMS-QQ-C-320. A ferroxyl test was completed by soaking filter paper in ferroxyl solution and heating to 180-200 degrees F. The paper was then placed over the entire plated area on each piece for ten minutes. Blue markings occurred on the filter paper as a result of the reaction of the base metal with the solution. These markings are indicative of porosity.

All the pieces plated in the baseline test passed the porosity test.

(e) Plating Hardness Test

Hardness testing was completed in accordance with ASTM B578. A Vicker's indenter with a 100 gram load was used. The minimum acceptable hardness as per the standard was 700 HV. The average hardness for all samples was above the 700 HV minimum (Table G-6). Additionally, there were no individual hardness readings below 700 HV.

Table G-6. Results of the hardness test for pieces plated with 4" PRD-EL1 fluid.

Part No.	4" PRD Hardness (HV)
145H7359-1	885.87
12304148	858.6
12286191	894.4
12284387	835.07
Coupon	893

Figure G-18 shows a plot of the hardness results.

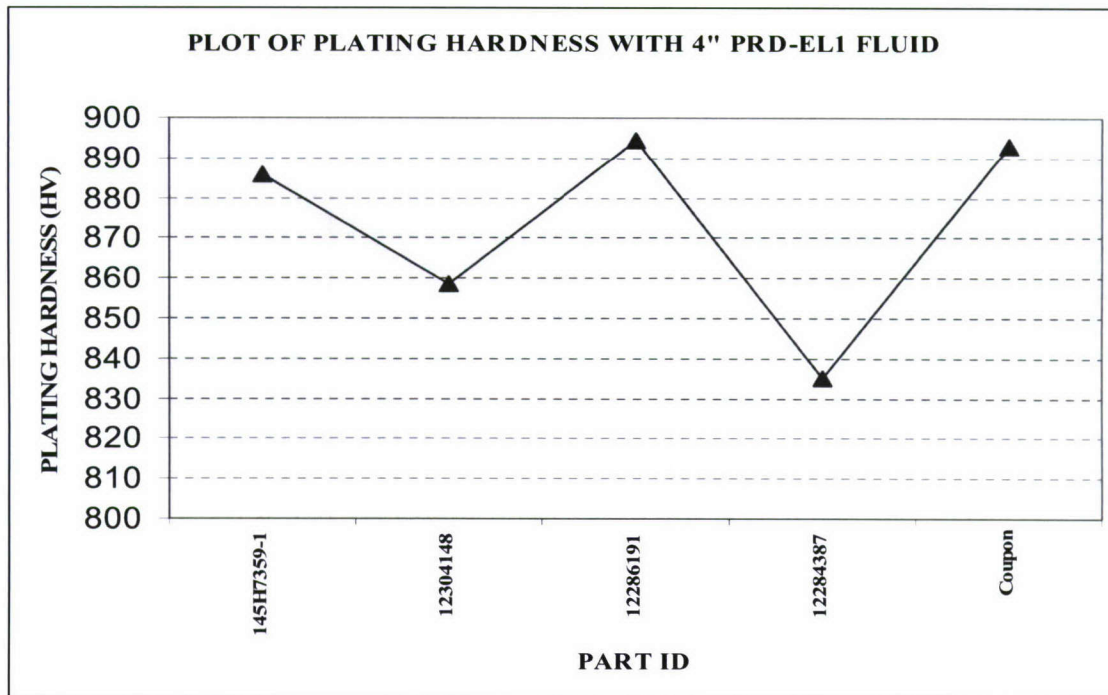


Figure G-18. Plot of hardness results with 4" PRD-EL1 liquid.

The LP Shaft (Part ID 12286191-1) had the highest hardness value and the Spur Gear (Part ID 12284387) had the lowest plating hardness. It is difficult to say whether these differences are significant without knowing the standard deviations of the actual data.

CONCLUSION

The majority of the parts plated with PRD-EL1 fluid passed all qualitative (adhesion, quality, porosity) requirements. Parts which failed some of the tests could be attributed to the following causes:

Bad contact at cathode; Current flow was significantly lower than at other cathodes, resulting in less plating thickness.

Lack of mixing, especially near the tank wall; Parts located at or near the center of the tank exhibited higher plating thicknesses than parts near the ends of the tank.

Shadowing effect, which occurred with coupons located between large parts, which resulted in low plating thicknesses and failures in adhesion testing. The edges of the coupons plated creating finger type projections, which was due to higher current densities (amperes/area) at the edges of the coupons.

Inadequate surface cleaning prior to plating or incomplete removal of wax during heating and reverse etching prior to forward plating operation

High concentration of ferrous oxide (visible rust) which was stirred by chromium liquid recycle through the sparger, located at the bottom of the tank.

Figure G-19 summarizes the main causes of inadequate plating thickness for the pieces plated in the Baseline (Run A), Run B with 2" PRD-EL1 fluid, and Run C with 4" of PRD-EL1 fluid. Using a plating thickness of 0.01 inches as acceptable and discarding data for all pieces with plating thicknesses less than 0.01 inches, a subset of the total dataset was obtained, as given in Table G-7.

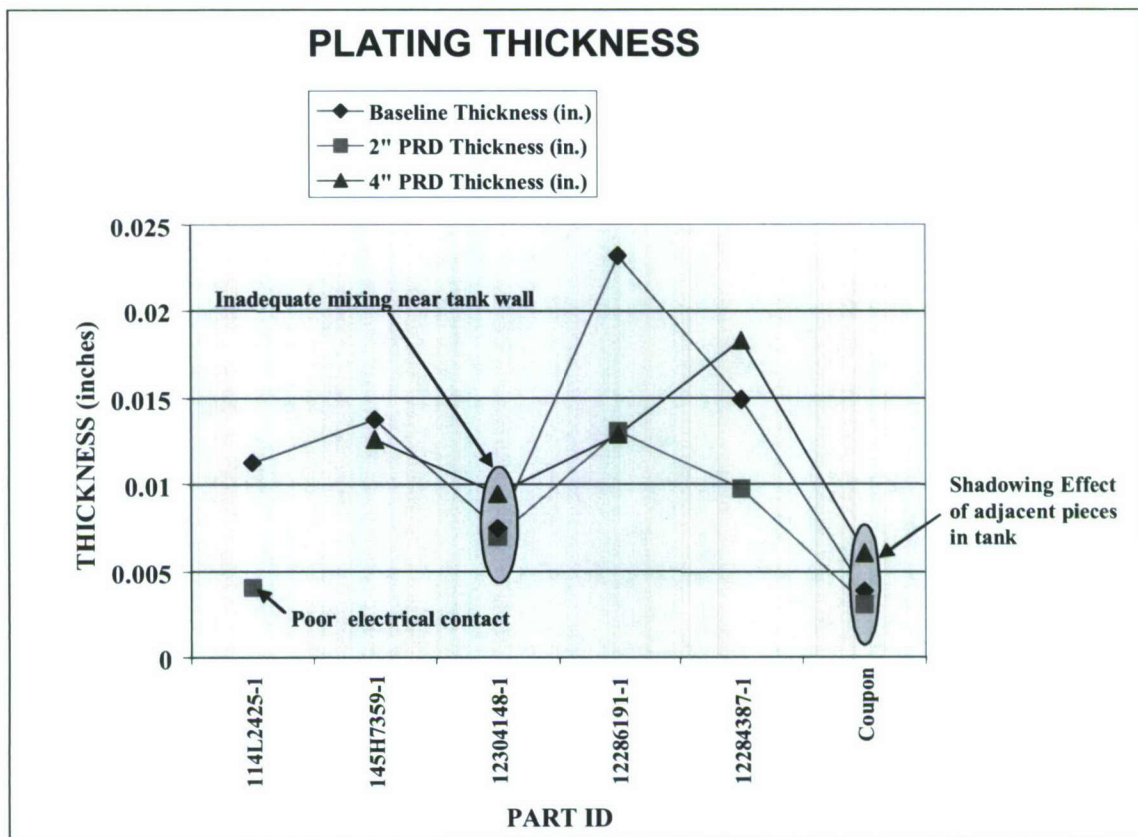


Figure G-19. Summary of causes for inadequate plating thickness.

Table G-7. Reduced subset of plating thickness and hardness data.

PART ID	PLATING THICKNESS (inches)	PLATING HARDNESS (HV)
114L2425-1	0.0113	906.90
145H7359-1	0.0126	885.87
12286191-1	0.0129	894.40
12286191-1	0.0131	885.47
145H7359-1	0.0137	921.33
12284387-1	0.0149	885.33
12284387-1	0.0183	835.07
12286191-1	0.0232	877.07

The average plating thickness for the reduced dataset, summarized in Table G-7 is 0.015 inches and the standard deviation is 0.004 inches. Since the plating thicknesses for all the parts in this reduced dataset fall within two standard deviations of the average, it can be concluded that there is no statistically significant difference in plating thickness between all the parts included in the reduced dataset. Hence, the presence of PRD-EL1, or its different layer thickness (2" and 4"), does not statistically impact plating thicknesses, when compared to the conventional plating process (no PRD-EL1 blanket).

The average plating hardness for the reduced dataset is 886.43 HV and the standard deviation is 25.15 HV. All hardness measurements also fall within two standard deviations of the average, indicating that there is no statistical difference between the hardness values for the tests included in the reduced dataset. Hence, the presence of PRD-EL1, or its different layer thickness (2" and 4"), does not statistically impact plating hardness, when compared to the conventional plating process (no PRD-EL1 blanket).

REFERENCES

1. Testing Results, December 9, 2002 Preliminary Report by Ashley Fiegel
2. Field Observation Notes – PRD Tech, Inc., August 29, 2002.
3. Test Protocol, Air Pollution Management Study, No. 43-EL-5166-03.
4. Zero-Emission Process for Electroplating Operation

APPENDIX A

DESCRIPTION OF THE ZERO-EMISSION PROCESS

FOR ELECTROPLATING OPERATIONS

ZERO-EMISSION PROCESS FOR ELECTROPLATING OPERATIONS

Hard chromium plating is extensively used in the industry to improve the wear characteristics of metal parts. It is also used for decorative purposes. EPA has identified hexavalent chromium, used in chromium plating, as one of the 17 high-priority toxic chemicals and is known to be a human carcinogen^{1,2}. It has been classified by the U.S. EPA as a Group A carcinogen³. Inhalation is the major exposure pathway for Cr⁶⁺. Workplace exposure to Cr⁶⁺ has been associated with a number of sources – metal plating, spray painting, welding, tanning, and abrasive blasting operations.

Regulation of bath temperature and mixing of the plating solution are essential for successful electroplating. Reject rate of parts increases due to non-uniformity of temperature in the plating bath and this increases waste generation. In some cases, temperature control is achieved by using heat ex-changer coils with cooling water, and the generation of gases due to electrolysis of water during electroplating mixes the bath fluid. In other cases, air sparging is used to cool the bath during electroplating and mix the fluid in the bath. For both cases, aerosols are produced when the gas bubbles break at the bath surface, and these aerosols are responsible for the chromium emissions.

Recently, a new Zero-Emission Electroplating process has been developed by PRD Tech, Inc. which can be retrofitted in existing chromium electroplating baths, and result in substantially reduced emission of chromium aerosols during electroplating. The process can be implemented in two ways: (1) A floating liquid layer of PRD-EL1, which is recirculated through the bath using an adjustable skimmer and external diaphragm pump, as shown in Figure G-21; and (2) Floating liquid layer of PRD-EL1, with an external mounted pump which recirculates the chromium liquid through a sparger with cooling achieved by a heat exchanger coil, as shown in Figure G-22. In both implementations, a proprietary liquid, PRD-EL1 is used to create a floating liquid blanket layer on the chromium bath. The liquid is colorless, transparent, physiologically inert, non-volatile, immiscible with the chromic acid solution, electrically non-conductive, with density less than water. These properties allow the PRD-EL1 to float as a blanket on the chromium liquid, without creating any

emissions on its own, and preventing the emission of chromium aerosols from the bath. Parts to be plated can be placed in the bath by passing through the liquid blanket and plated parts can be withdrawn from the bath. Rinsing of the part above the bath allows any liquid blanket attaching to the part's surfaces to be returned back to the plating bath.

The main advantages of using the PRD-EL1 liquid blanket technology are:

- (1) substantial reduction of chromium aerosols, which cause chromium exposure to working personnel and result in chromium emissions;
- (2) due to reductions in chromium emissions from the tank, venting rates from the plating shop by exhaust fans can be reduced; this causes substantial energy savings especially for plating shops in cold climate locations, where air heating costs can be a substantial fraction of the shop's operating budget;
- (3) temperature in the bath is maintained either by the cooling coils in the bath or by cooling of the recirculating PRD-EL1 liquid, and mixing in the tank is accomplished by either recirculation and sparging of PRD-EL1 within the tank or by recirculation and sparging of the chromium solution; and
- (4) elimination of fume suppressant costs.

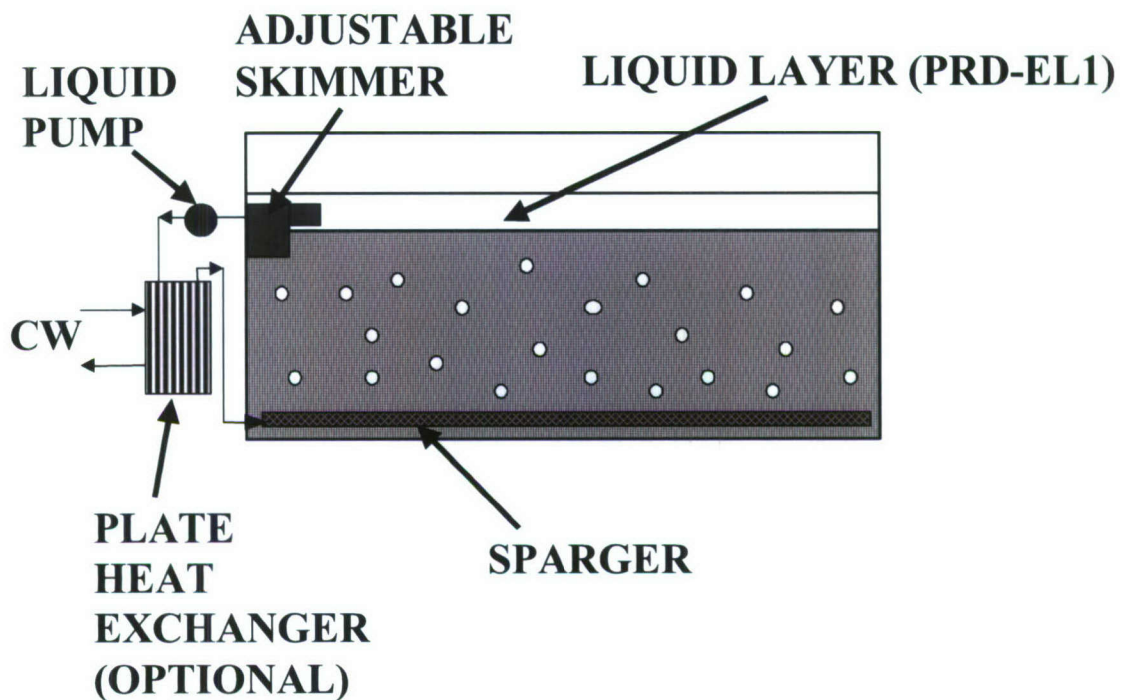


Figure G-20. Schematic of the Zero-Emission Process with Blanket Liquid Recirculation.

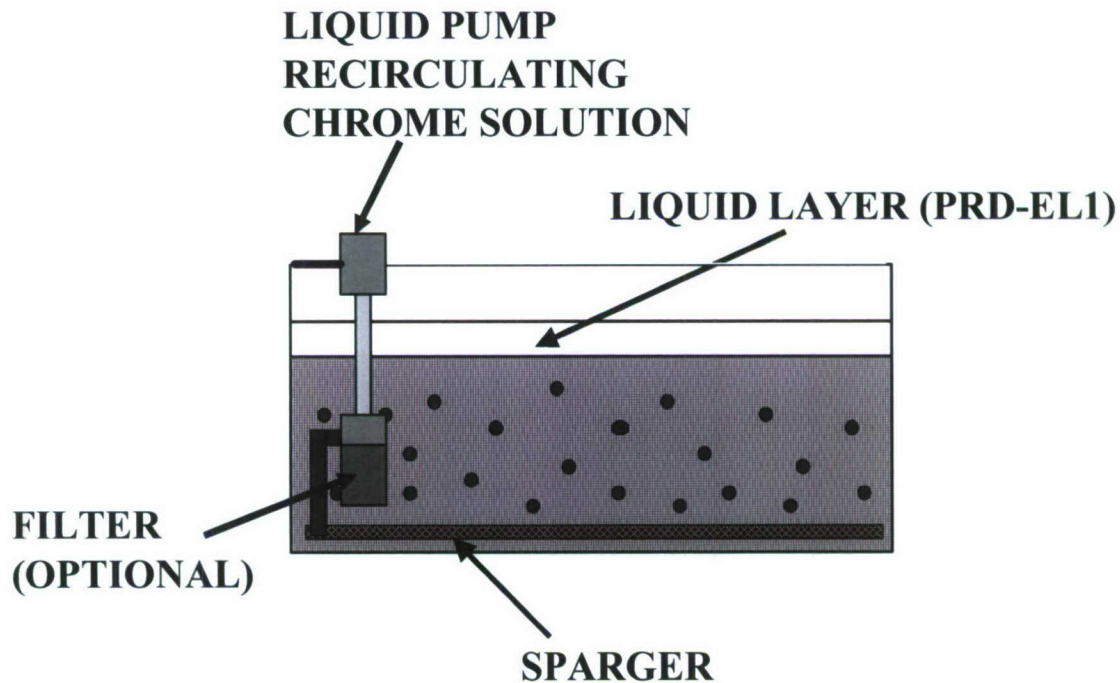


Figure G-21. Schematic of the Zero-Emission Process with Chromium Liquid Recirculation.

Appendix H: PRD, Inc. Report on Phase II Results

DATA ANALYSIS FOR PRD-EL1 DEMONSTRATION TEST – PHASE II – AIR EMISSION TESTING

Submitted to:

Dr. Steve W. Maloney
U.S. Army Engineering Research and
Development Center
Champaign, IL 61826-3482

Submitted by:

Dr. Rakesh Govind
Director of Engineering, PRD Tech, Inc.

and

Professor of Chemical Engineering
University of Cincinnati
Tel: (859) 578-8010
Fax: (859) 578-8015
Email: rgovind837@aol.com

August 2003

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ABSTRACT

PRD Tech, Inc. has developed a Zero-Emission process, which uses a proprietary liquid blanket, de-signed as PRD-EL1, which floats on the chromic acid liquid and reduces emission of chromic acid aerosols, emitted during hard chromium plating operation. Testing was conducted to evaluate the effect, if any, of the PRD-EL1 fluid on the extent of air emissions during electroplating. Testing was conducted at the Anniston Army Depot (ANAD), Building 114, Alabama. Plating was conducted on several components and test coupons using Line 2, while concurrent production was being con-ducted in Line 1, as scheduled. Line 2 had a total of four chromium vats. For this demonstration only vats 12A and 12B were used.

The fumes from all chromium vats, were pulled through one duct using an induced draft fan, which exhausted through an entrainment separator to a 38-inch inside diameter stack. For this demonstra-tion test, the Alabama Department of Envi-ronmental Management (ADEM) had allowed the separa-tor to be removed from the system. Sampling was conducted through two 4-inch ID ports, located at right an-gles to each other and located 3 duct diameters downstream and 1.4 duct diameters upstream from the nearest flow disturbances. Using EPA Method, a velocity trans-verse of 24 sampling points (12 per transverse) was conducted using a pitot tube/thermocouple assembly. Details of the sampling procedure are presented in the Appendix Report.

In addition to stack sampling, Total Suspended Particulate (TSP) samplers were used to determine the chromium levels in the indoor atmosphere. The West sam-pler was located approximately six feet west of chromium plating vat 12 B. The East Sampler was located approximately 12 feet east of chromium plating vat 12B.

Tests were conducted with 4 inches, 2 inches and no PRD liquid. The average total chromium con-centrations for each test series, as measured per USEPA Method 306, was below the 0.015 mg/dscm National Emission Standards for Hazardous Air Pollutants (NESHAP) – Chromium standard. There was substantial reduction in chromium emissions due to the presence of PRD liquid, although the effect of liquid blanket thickness could not be quantified due to emissions from other tanks that were operating without the PRD liquid.

INTRODUCTION

Chromium plating of machinery parts produces a surface coating that helps reduce wear and corrosion. The military uses these processes as a cheap and effective way to combat the wear and corrosion of parts suffer during usage. The problem with chromium plating is the emission of a fine aero-sol, during the plating process. Once in the atmosphere, the aerosol forms chromic acid. Hexavalent chromium has long been known to be a carcinogen and cause of perforated nasal passages, skin rashes and other medical problems. These emissions are controlled by capturing the aerosols at the surface with airflow above the vat, which enters an exhaust duct and exits from a stack outside the building.

PRD Tech, Inc. has developed a proprietary Zero-Emission process (Appendix 1) which uses an immiscible liquid that covers the top of the chromium bath during the plating process. This liquid, designated as PRD-EL1, prevents the aerosols of chromium from reaching the atmosphere by trapping the bubbles before they reach the liquid-air interface. If successful, this process may replace expensive scrubber technology currently used to deal with emissions.

Testing was conducted to evaluate the effect, if any, of the PRD Tech proprietary fluid on the quality of chromium plating. Testing was conducted in Building 114 on the Anniston Army Depot (ANAD), which houses the depot's metal finishing operations. Vats 12A and 12B in Line 2 were used for the test.

The plating conditions used were as follows:

Voltage:	Vat 12A: 7.0 V; Vat 12B: 3.8 – 4.0 V
Amperage:	Vat 12A: 1,100 amps; Vat 12B: 300 amps
Temperature:	Vat 12A:123oF; Vat 12B: 122oF (hand held thermometer)
Plating time:	12 hours
Reverse Etching:	Before plating at 4 volts, 225-230 amps

Plating was conducted on test coupons, Adapter Assembly, and LP shaft.

The experimental design and components plated were as follows (Table H-1).

Table H-1. Reduced subset of plating thickness and hardness data.

Test Run Series	Part Description	Comments
1 (Runs 1, 2, 3)	Test Coupons, Adapter, LP Shaft	<p>4" PRD liquid thickness Chromium plating occurring in Vat on line 2, which is not used for demonstration. Tank 8 in line 1 was also operating, plating two small parts. Another concern was that Tank was about 25 ft south of the background air sampler, and emissions from this tank move directly across the suction registers of Tanks 15 and 16 (mid-suction registers) in Line 2, and these emissions are not accounted by the background air sampler. This may skew the results from tanks 12A and 12B.</p> <p>that Tank 8 was about 25 ft</p>
2 (Runs 4, 5, 6)	Test Coupons, Adapter, LP Shaft	<p>2" PRD liquid thickness. Tank 10 (Line 1) was not plating. Tank 2 was plating. The lower suction registers of Tank 16 (Line 2) were covered; however the upper registers were still open, leaving the possibility of cross-contamination into Line 2 duct from Tank 8 emissions. The first run in this series was started at 8:20 am; however Vat 12B was not started until 9:00 am.</p>
3 (Runs 7, 8, 9)	Test Coupons, Adapter, LP Shaft	<p>No PRD liquid; there was some leftover PRD liquid that could not be removed from the chromium solution surface. The large vat at the end of Line 1 was plating, and potential fumes from this operation, if introduced into Line 2 exhaust, could bias results.</p>

TESTS CONDUCTED

The following tests were conducted to evaluate the quality of plating:

I. Quality of Plating: Visual observations of the plated parts before thermal annealing

II. Air Sampling: (a) USEPA RMs 1-4 were used to verify sampling points, conduct velocity transverse and cyclonic flow checks, and to determine moisture and stack gas content.

(b) USEPA 306 for Total Chromium

(c) Total Suspended Particulate (TSP)

2 hour samples were collected from two locations.

RESULTS AND DISCUSSION

Test Run 1: 4" of PRD Liquid thickness in Vats 12A and 12B (Table H-2).

Table H-2. Test Run 1: 4" of PRD liquid in Vats 12A and 12B.

Run Number	Total Amperage (amps)	Total Cr in Stack (mg/dscm)	TSP –West (mg/m3)	TSP-East (mg/m3)
Run 1	1,300	0.016	0.125	0.067
Run 2	1,400	0.009	0.010	0.005
Run 3	1,400	0.007	0.004	0.002

Tank 12A had noticeable blobs floating on the surface and also at the interfacial layer of PRD fluid and the chromium acid solution. Tank 12B did not have any such blobs.

All the parts removed after plating looked fairly good. There were no visible streaks, water breaks, etc. The parts were sent for baking next day in the morning.

Clearly, Run 1 results from the above table are significantly different than the results for Runs 2 and 3. Neglecting the results of Run 1, since at this time, emissions from Tank 8 were being collected by the suction registers of Tanks 15 and 16 into the line 2 stack, we get the following average results:

Total Amperage: 1,400 amps
 Total Cr in Stack (mg/dscm): 0.008
 TSP – West (mg/m³): 0.007
 TSP – East (mg/m³): 0.0035

Test Run 2: 2" of PRD Liquid thickness in Vats 12A and 12B (Table H-3).

Table H-3. Test Run 2: 2" PRD liquid thickness in Vats 12A and 12B.

Run Number	Total Amperage (amps)	Total Cr in Stack (mg/dscm)	TSP –West (mg/m ³)	TSP-East (mg/m ³)
Run 4	1,400	0.011	0.020	0.006
Run 5	1,400	0.004	0.019	0.005
Run 6	1,400	0.008	0.009	0.004

Tank 16 (Line 2) is covered (lower suction registers only). Upper registers are still open, which gives the possibility of cross contamination.

The plating was fair, especially on the test coupons. The Adapter assembly had a slight water mark. During this run, the voltage fluctuated (3.7 – 4.7 V) and current (50 – 250 amps) throughout the plating run. The contacts were checked and found to be fine.

The average emissions were as follows:

Total Amperage: 1,400 amps
 Total Cr in Stack (mg/dscm): 0.008
 TSP-West (mg/m³): 0.016
 TSP – East (mg/m³): 0.005

Test Run 3: No PRD Liquid in Vats 12A and 12B (Table I-4).

Table H-4. Test Run 3: No PRD liquid in Vats 12A and 12B.

Run Number	Total Amperage (amps)	Total Cr in Stack (mg/dscm)	TSP –West (mg/m ³)	TSP-East (mg/m ³)
Run 7	1,400	0.013	0.130	0.027
Run 8	1,400	0.013	0.098	0.021
Run 9	1,400	0.015	0.071	0.011

Tank 10 in line 1 is running. Tank 8 is expected to run for 20 hours. Tank 16 is covered.

All of PRD liquid could not be removed. Further, air flow in the large tank was not even, since the pipe was broken in the middle.

Total Amperage:	1,400 amps
Total Cr in Stack (mg/dscm):	0.007
TSP-West (mg/m ³):	0.004
TSP – East (mg/m ³):	0.002

The average results are plotted below (Figure H-1). Clearly, the emissions in the stack and in ambient air were higher when no PRD liquid was present in Vats 12A and Vats 12B. It is not known what the actual reductions for tanks 12A and 12B were, since the stack measurements included other operating tanks also. There was also crossover contamination from line 1, as noted earlier. The TSP sampling showed that ambient air samples fluctuated due to other operations in Line 1. TSP-West was always higher than TSP-East, due to the effects from other operating tanks in Line 1.

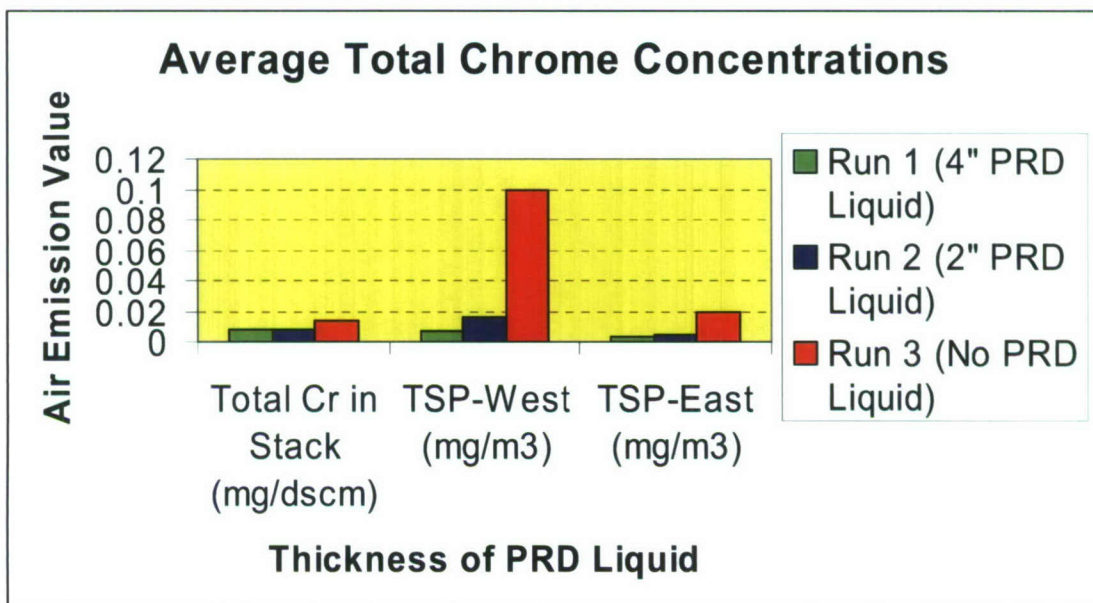


Figure H-1. Average chromium concentrations for the runs.

Appendix I: CHPPM Report on Air Sampling

Appendix I is Volume 2 of this report.

Appendix J: Air Sampling Protocol

MCHB-TS-EAQ (40)

MEMORANDUM FOR Commander, USACERL (W-ERDC-CERL-IL/Dr. Steve W. Maloney), U.S. Army Engineering Research and Development Center, Champaign, IL 61826-3482

SUBJECT: Test Protocol: Air Pollution Management Study No. 43-EL-5116-03, Chromium Plating Finishing Complex, Building 114, Anniston Army Depot, Alabama

1. Preliminary site visit by Mr. Robert Wishart, Mr. Tim Hilyard, and Mr. Bret Mower, this Center, Mr. Tony Pollard and Mr. Jeremy Turner, Chromium Plating Finishing Complex, 3 June 2002, subject: Zero Chromium Emission Process.
2. Per reference, a test protocol for the assessment of the emissions for the above exhaust ducts has been prepared and is enclosed for your review.
3. Our point of contact is Mr. Tim Hilyard, this Center, DSN 584-2509/3500 or commercial (410) 436-2509/3500.

FOR THE COMMANDER:

Encl JAMES D. WOOD, P.E.
 Program Manager
 Air Quality Surveillance

CF:

CDR, ANAD, ATTN: AMSTA-AN-PECE (Tony Pollard)

TEST PROTOCOL

AIR POLLUTION MANAGEMENT STUDY

NO. 43-EL-5166-03

CHROMIUM PLATING FINISHING COMPLEX

BUILDING 114

ANNISTON ARMY DEPOT, ALABAMA

Distribution limited to U.S. Government agencies only; protection of privileged information evaluating another command; Jan 03.
Requests for this document must be referred to Commander, USACERL (W-ERDC-CERL-IL), U.S. Army Engineering Research and Development Center, Champaign, IL 61826-3482

LIST OF ACRONYMS

ANAD	Anniston Army Depot
Co	company
Cr	chromium
GFAAS	graphite furnace atomic absorption spectroscopy
in	inches
Inc	incorporated
mL	milliliter
N ₂	nitrogen
NaOH	sodium hydroxide
O ₂	oxygen
PRD Tech.	Process, Research, and Development Technologies
PW	probe wash
RM	Reference Method
QA/QC	quality assurance/quality control
USACERL	U.S. Army Construction Engineering Research Laboratory
USACHPPM	U.S. Army Center for Health Promotion and Preventative Medicine
USEPA	U.S. Environmental Protection Agency
VOCs	volatile organic compounds
°	degree

TEST PROTOCOL
AIR POLLUTION MANAGEMENT STUDY 43-EL-5166-03
CHROMIUM PLATING FINISHING COMPLEX
BUILDING 114
ANNISTON ARMY DEPOT, ALABAMA

1. REFERENCES. See Appendix A for a list of references.
2. PURPOSE. The purpose of this assessment is to determine the effectiveness of the PRD Zero-Emission Process in removing chromium (Cr) emissions from a full-scale chromium plating operation. Volatile organic compounds (VOCs) may also be measured to determine whether the Process Research and Development Technologies (PRD Tech. Inc.) proprietary immiscible liquid emits any VOCs into the atmosphere.
3. GENERAL.
 - a. Background. Chromium plating of machinery parts produces a surface coating that helps reduce wear and corrosion. The military uses these processes as a cheap and effective way to combat the wear and corrosion of parts suffer during usage. The problem with chromium plating is the emission of a fine aerosol, during the plating process. Once in the atmosphere, the aerosol forms chromic acid. Cr has long been known to be a carcinogen and a cause of perforated nasal passages, skin rashes and other medical problems. These emissions are controlled by capturing the aerosols at the surface with airflow above the vat, which enter an exhaust duct and exit from a stack outside the building. PRD Tech, Inc. has developed a proprietary immiscible liquid that covers the top of the chromium bath during the plating process. This liquid prevents the aerosols of chromium from reaching the atmosphere by trapping the bubbles before they reach the liquid-air interface. If successful, this process may replace expensive scrubber technology currently used to deal with emissions.
 - b. Facility Description. The Zero-Emission demonstration will take place in Building 114 on the Anniston Army Depot (ANAD). This building houses the depot's metal finishing operations. Line 2 will be used in this demonstration to allow for continued production on Line 1. Line 2 has a total of four chromium vats, all of which will be used in this demonstration.
 - c. Exhaust System Description. The exhausts from all the chromium vats join into one duct. The exhaust exits out of a stack on the side of the building. The stack has a diameter of 36 inches (in).

**Use of trademark names does not imply endorsement by the U.S. Army
tended only to assist in identification of a specific product.**

4. SAMPLING AND MONITORING REQUIREMENTS. The ANAD and U.S. Army Construction Engineering Research Laboratory (USACERL) have requested that the U.S. Army Center for Health Promotion and Preventative Medicine (USACHPPM) perform the emission testing for the Zero-Emission demonstration. Sampling will be conducted using United States Environmental Protection Agency (USEPA) Reference Method (RM) 306 (reference 1) to determine the total Cr emissions. If conducted, the USEPA Compendium Method TO-14A (reference 3) will be used to measure VOCs that might be emitted by the PRD Tech, Inc. proprietary immiscible liquid. At this time a final determination concerning the VOC sampling has not been made.
5. TEST CONDITIONS. All requirements for a compliance test will be followed during this demonstration.
6. SAMPLING EQUIPMENT AND PROCEDURES.
 - a. Stack Sampling Location. For the test, the existing ports on the line 2 exhaust stack will be used for the measurement of Cr emissions.
 - b. Ambient Sampling Location. A TO-14A SUMMA canister will be used for determination of VOC levels that are emitted. The TO-14A will be inserted in the stack across from the existing ports.
 - c. Sampling Method.

(1) Chromium Sampling. The USEPA RM 306 (reference 1) will be used to measure the Cr emissions being released to the atmosphere at the stack. The train configuration is as follows:

- Pyrex® sample nozzle
- Teflon® union
- Pyrex lined probe sheath assembly
- Teflon® flex line
- 90° elbow
- Impinger No. 1-100 mL 0.1 N NaOH solution
- 180° glass connector
- Impinger No. 2-100 mL 0.1 N NaOH solution
- 180° glass connector
- Impinger No. 3-dry
- 180° glass connector
- Impinger No. 4-silica gel

® Pyrex is registered trademark of Corning Glass Works, Houghton Park, Corning, New York
® Teflon is a registered trademark of E. I. DuPont de Nemours & Co., Inc., Wilmington Delaware

S-type pitot tubes and thermocouples will be attached to the sampling probe. The pitot tubes will be 0.75-in. from the probe nozzle, and the thermocouples will be placed to eliminate any disturbance in the velocity measurements. The probe will be attached to a sample box containing the impinger train by a teflon flex line. The impingers will be packed in an ice bath to cool the gas and to remove the moisture from the gas sample. The sample box will be connected to an umbilical cord, which contains the vacuum line, pitot lines, electrical connections and thermocouple wires. The meter box has a calibrated dry gas meter and calibrated orifice. A vacuum pump will be used to draw the sample through the sampling equipment. Two manometers, mounted on the meter box, will measure the velocity pressure in the stack and the pressure differential across the meter box orifice.

(2) VOC Sampling. If conducted, the VOCs will be collected and analyzed according to USEPA Compendium Method TO-14A. The TO-14A canister sampling modes that will be employed for this test are pressurized. Pressurized sampling requires an additional pump to provide positive pressure to the sample canister. A sample of the stack gas will be drawn through a sampling train comprised of components that regulate the rate and duration of sampling into specially prepared canister.

d. Sampling Procedures.

(1) Cr Sampling. The USEPA RM 306 sampling train will be operated isokinetically. Sampling will be performed by controlling the sampling flow rates, so the velocities of the gases entering the sampling nozzle are equal (within ± 10 percent) to those of the undisturbed stack gas stream at the sampling points. Since this is not a fuel burning source, gas composition will be considered as ambient air (i.e., 79 percent N₂, and 21 percent O₂).

(2) VOC Sampling. If conducted, the USEPA Compendium Method TO-14A sampling will be operated at a constant collection rate. The canisters will be pressurized via a separate pump to increase the volume of the gas collected. The anticipated sample volume of the TO-14A canisters will be approximately 12 liters.

e. Sample Methods. In accordance with RM 1 (reference 1), a preliminary velocity, temperature and cyclonic flow traverse will be conducted. Three valid 2-hour runs will be performed for each test series (no foam, 2-inches of foam and 4-inches of PRD foam) in accordance with USEPA RM 306 (Figure H-2). Table H-5 is a summary of the sampling parameters to be monitored and evaluated.

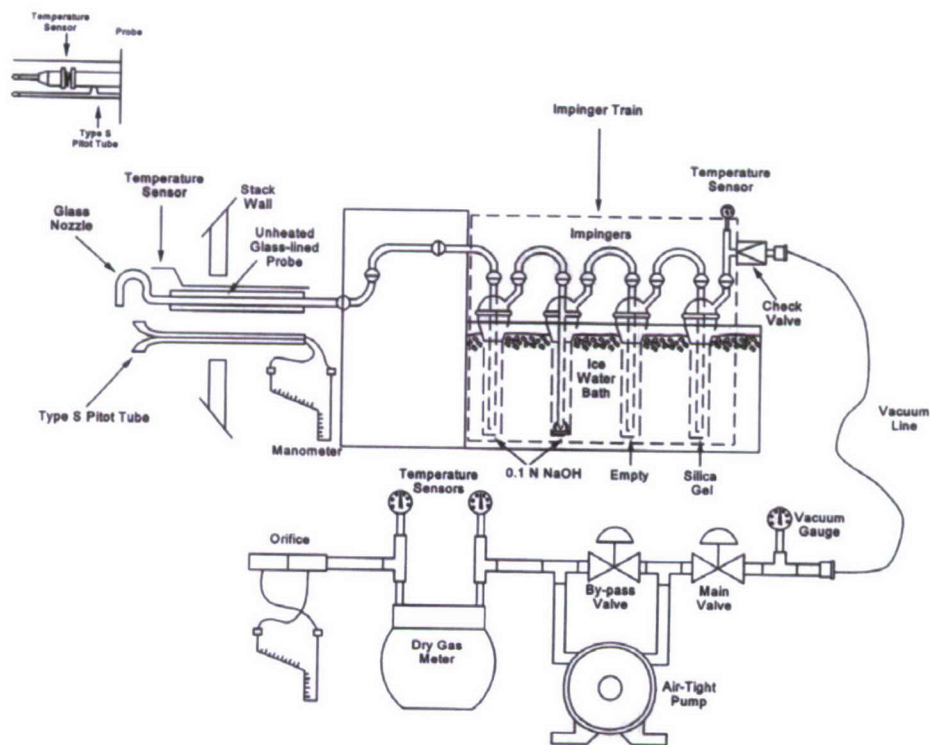


Figure J-1. USEPA RM 306 sampling train.

Impinger Contents

Impinger 1 – 100 mL 0.1 N NaOH

Impinger 2 - 100 mL 0.1 N NaOH

Impinger 3 – Initially Dry

Impinger 4 – Silica Gel

Table J-1. Sample parameter summary.

PARAMETER	METHOD	RESPONSIBLE ORGANIZATION
Cr Emissions	RM 306	USACHPPM
Stack Temperature	RM 5	USACHPPM
Stack Gas Flow Rate	RM 2	USACHPPM
Stack Gas Moisture	RM 4	USACHPPM
VOCs	TO-14A	USACHPPM

7. TEST PLAN

a. Chromium Plating Process. During the test, the chromium plating line will be operated under normal conditions. Dummy plates will be used to determine whether the PRD Tech, Inc. fluid has any effect on the efficiency of the chromium plating. Some typical parts may also be used in the plating process.

b. Exhaust System. The exhaust system will be operated under normal operating conditions with the exception that the demister pads will be removed from the system.

c. Sampling Summary. The test on the exhaust stack will consist of three series of three sampling runs in each series. During the first series the plating vats will be run with no PRD Tech, Inc. fluid on the vats. The next two series will be with 2-inches and 4-inches of PRD fluid respectively on top of the plating solution. During each run, moisture and temperature will be continuously monitored to determine the moisture content and temperature of the stack gas in the ducts. Table J-6 consists of a summary of the sampling that will be conducted.

d. Sampling Locations. For the test, the existing ports on the Line 2 exhaust stack will be used for the RM 306 train. These ports meet the requirements of USEPA RM 1. A total of 12 traverse points will be used. If conducted, the TO-14A sampling will be performed on a separate port of the stack opposite to the existing ports.

e. Sampling Duration. As indicated in paragraph 6.d, the sampling run duration will be 2 hours.

Table J-2. Sampling summary.

FLUID LEVELS	RUN DURATION	COLLECTION FREQUENCY	SAMPLING METHOD	ANALYSIS PARAMETER
No fluid	2 Hr	Continuous Continuous Continuous Continuous Continuous	RM 306 TO-14A RM 5 RM 4 RM 2	Cr VOCs Temperature Moisture Stack Gas volumetric flow rate
2 inches	2 Hr	Continuous Continuous Continuous Continuous Continuous	RM 306 TO-14A RM 5 RM 4 RM 2	Cr VOCs Temperature Moisture Stack Gas volumetric flow rate

f. Sample Recovery. The USEPA RM 306 train will be used to determine the total chromium emission rate. The train will be recovered as follows: Sample 1 (Probe Wash and Impingers). Measure the volume of the first, second, and third impingers, then quantitatively transfer the liquid into a labeled sample container (Container 1). Rinse the probe nozzle, probe liner, flex line, the three impingers and connecting glassware with approximately 200 to 300 mL of 0.1 N NaOH. This rinse will be added to Container 1. Next, place a signed and dated sample custody seal over the lid and top of jar to ensure the lid is not removed prior to the analytical lab receiving the sample.

g. Sample Analysis.

(1) Stack Gas Analysis.

(a) Stack Gas Composition. The stack gas will be considered ambient air, since there is no combustion process.

(b) Stack Gas Moisture Determination. Moisture is collected from the stack gas by the four impingers in the sampling train. The total amount of moisture is determined by weighing the impingers and their contents before and after each run. The difference gained after the run is the amount, in grams, of moisture collected. The weight gained in grams is equal to the volume of the moisture in mL. All impingers will be weighed on a top-loading balance accurate to 0.01 grams.

(2) Train Sample Analysis. The analytical procedures to be used to analyze the train samples generated during this test are summarized in Table J-7. The Cr analysis will be done using graphite furnace atomic adsorption spectroscopy (GFAAS) (reference 4) per Method 306 procedures.

8. QUALITY ASSURANCE/QUALITY CONTROL (QA/QC).

a. General. The QA/QC procedures for the sampling and analytical methods to be used will consist of pre-test and post-test calibrations of sampling equipment, analysis of blank samples and of all reagents and collection medias.

Table J-3. Analytical procedures summary.

COMPONENT	PARAMETER	TECHNIQUE	ANALYSIS
Method 306 Train			
Impinger 1-4	Moisture	Gravimetric	Analytical Balance
PW/Impingers	Total Cr	GFAAS	SW-846 Method 7191*

* Reference 4

b. Sampling Equipment. Prior to and immediately following the test, all sampling equipment will be calibrated using the procedures outlined in reference 2. Calibrations of the probe nozzles, pitot tube alignment, dry gas meters, thermometers, and thermocouple/pyrometer assemblies will be conducted. Table J-8 summarizes these equipment calibrations.

Table J-4. Calibration procedures summary.

DEVICE	METHOD/STANDARD	REFERENCE
Meter Box Orifice	Wet Test Meter	APTD-0576*
Dry-Gas Meter	Wet Tester Meter	APTD-0576*
Pyrometer	NBS Reference Pyrometer	USEPA RM 5**
Pitot Tube	Geometry	USEPA RM 2**
Thermometer/Thermocouple	Reference Pyrometer	USEPA RM 2**
Nozzle	Micrometer	USEPA RM 5**

* Reference 2

** Reference 1

c. Analysis.

(1) General. All calibration standards and reagents will be of the highest purity practical for analysis and will meet USEPA RM requirements.

(2) Chromium Analysis.

(a) Field Blanks. Field blanks of all collection and recovery media will be taken for analysis. The field reagent blank is used to assess possible contamination resulting from processing the sample.

(b) Laboratory Analysis. All QA/QC procedures of Method 7191 of SW-846 (reference 4) will be performed.

9. TENTATIVE TEST SCHEDULE. A tentative schedule for the subject emission assessment is listed below. This schedule may be used by the installation to prepare the proper support for the emission assessment.

- a. March 2 (Sunday): Travel to ANAD, prepare test site, set up mobile laboratory, and set up sampling sites. Conduct preliminary stack testing (cyclonic flow and stack gas velocity traverse).
- b. March 3 (Monday): Conduct Runs 1 and 2 (No fluid) and recover samples.
- c. March 4 (Tuesday): Conduct Run 3, change fluid level in the vats. Conduct Run 4 (2-in fluid) and recover samples.
- d. March 5 (Wednesday): Conduct Runs 5 and 6 (2-in fluid) and recover samples. Change fluid level in the vats.
- e. March 6 (Thursday): Conduct Runs 7 and 8 (4-in fluid) and recover samples. Pack equipment.
- f. March 7 (Friday): Conduct Run 9 and recover samples. Pack equipment.
- g. March 8 (Saturday): Contingency Day. Travel to Aberdeen Proving Ground.

10. SAFETY.

- a. General.

If any unsafe action or situation is observed, the USACHPPM project officer onsite will be informed at once. Under these circumstances, the survey personnel will be removed from the hazardous condition until it is considered safe to return. When the safety of a situation is uncertain, the ANAD Safety Officer will examine the situation and determine if it is safe to return to the work area. Survey personnel will not attempt to correct the situation by themselves if it would expose anyone to the hazard.

- b. Weather.

All personnel will protect themselves from weather or climate related injuries. The project officer may terminate the test if extreme weather conditions (heavy rain, high winds, thunderstorms, etc.) exist.

TIMOTHY D. HILYARD
Environmental Protection Specialist
Air Quality Surveillance Program

REVIEWED BY:

APPROVED BY:

MICHAEL S. PATTISON
Supervisor
Air Quality Surveillance
Program

JAMES D. WOOD, P.E.
Program Manager
Air Quality Surveillance

REFERENCES

1. 40 CFR 63, 2001 rev, National Emission Standards for Hazardous Air Pollutants for Stationary Source Categories, Appendix A, Test Methods.
2. USEPA Manual APTD-0576, March 1982, Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment.
3. USEPA Manual 625/R-96/0106, January 1999, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air.
4. USEPA Manual SW-846, Revision 1, December 1996, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Volume IA: Laboratory Ma

REPORT DOCUMENTATION PAGE

Form Approved

OMB No. 0704-0188

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing this collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden to Department of Defense, Washington Headquarters Services, Directorate for Information Operations and Reports (0704-0188), 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number. PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.

1. REPORT DATE (DD-MM-YYYY) 02-2008		2. REPORT TYPE Final		3. DATES COVERED (From - To)	
4. TITLE AND SUBTITLE Technology Demonstration of the Zero Emissions Chromium Electroplating System				5a. CONTRACT NUMBER	
				5b. GRANT NUMBER	
				5c. PROGRAM ELEMENT NUMBER	
6. AUTHOR(S) K. James Hay, Stephen W. Maloney, John J. Cannon, Max R. Phelps, and Jason Modrell				5d. PROJECT NUMBER	
				5e. TASK NUMBER 002	
				5f. WORK UNIT NUMBER CNE-B091	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) U.S. Army Engineer Research and Development Center (ERDC) Construction Engineering Research Laboratory (CERL) PO Box 9005 Champaign, IL 61826-9005				8. PERFORMING ORGANIZATION REPORT NUMBER ERDC/CERL TR-08-5, Vol. 1	
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) USEPA Facilities 26 West Martin Luther King Drive Cincinnati, OH 45268-0001				10. SPONSOR/MONITOR'S ACRONYM(S)	
				11. SPONSOR/MONITOR'S REPORT NUMBER(S)	
12. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release; distribution is unlimited.					
13. SUPPLEMENTARY NOTES					
14. ABSTRACT This report documents the demonstration of a technology developed by PRD, Inc, for control of chromium emissions during hard chromium electroplating, the Zero Emissions System. The technology involves placing a blanket of a proprietary fluid, called PRD-EL1, on top of the plating bath. This fluid blanket prevents the formation of aerosols, which is the mechanism by which chromium is emitted from the plating bath to the air. The majority of the testing was directed at demonstration of the effectiveness of chromium plating in the presence of the immiscible blanket. Testing was conducted at Benét Laboratories on coupons and actual parts from Army vehicles. The results indicate that PRD-EL1 may cause deleterious effects on the plating process, as some of the parts failed qualitative tests performed at Benét. However, some parts, which were plated without the fluid blanket present as a baseline control, also failed the tests. Air sampling results indicate that the presence of the PRD-EL1 fluid reduced the chromium emissions to below the standard and the indoor air concentration below the previously established exposure limit but near the new exposure limit.. Overall, the results indicate that the use of the PRD process would require additional testing before it could be accepted for use in Army production and maintenance operations.					
15. SUBJECT TERMS hazardous air pollutants (HAPs) air pollution plating emissions chromium					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT SAR	18. NUMBER OF PAGES 350	19a. NAME OF RESPONSIBLE PERSON
a. REPORT Unclassified	b. ABSTRACT Unclassified	c. THIS PAGE Unclassified			19b. TELEPHONE NUMBER (include area code)